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RANDOM STRUCTURES OF LAYER MINERALS AS ILLUSTRATED BY CRONSTEDITE ($2FeO \cdot Fe_2O_3 \cdot SiO_2 \cdot 2H_2O$). POSSIBLE IRON CONTENT OF KAOLIN

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Lack of replacement of aluminium in kaolin minerals by magnesium or iron is an extremely important factor in the formation of soil. Aluminium in muscovite can be replaced by magnesium and iron forming phlogopite and biotite. The iron present in micas and other ferro-magnesian minerals is expelled as oxides or hydrous oxides when weathering to kaolin takes place. Montmorillonite, nontronite and related minerals, on the other hand, which are more closely similar to micas than are the kaolin minerals, can retain iron as weathering proceeds.

The maximum amount of iron in the fourteen samples of kaolinite and anauxite described by Ross and Kerr[1] is 2.00 per cent Fe_2O_3 and only two of the samples, both of which were anauxites, contained more than 1.5 per cent Fe_2O_3. This corresponds to a maximum substitution of 4 per cent of Al^{+++} by Fe^{+++}. Thirteen analyses of halloysite,[2] six of dickite, and three of nacrite showed a maximum of 0.64 per cent Fe_2O_3 which corresponds to replacement of 1.00 per cent of Al^{+++} by Fe^{+++}. The magnesium content of these samples is also very low, not exceeding 0.35 per cent in unquestionable material.

Pauling[3] has shown that the structures of the kaolin minerals and chlorites are closely related to those of the micas. The four kaolin minerals, kaolinite, halloysite, nacrite and dickite, represent alternative methods of stacking aluminium-silicate layers having the composition $(OH)_4Al_2Si_2O_5$. Factors contributing to the polymorphism of these minerals have been discussed elsewhere.[4] Pauling stated, "The non-existence of a magnesium analog of kaolinite is accounted for by the large value

[1] Ross, C. S., and Kerr, P. F., The kaolin minerals: *Prof. Paper* **165E**, *U. S. Geological Survey* (1930).

[2] Ross, C. S., and Kerr, P. F., Halloysite and allophane: *Prof. Paper* **185G**, *U. S. Geological Survey* (1934–35).

[3] Pauling, L., *Proc. Nat. Acad. Sci.*, **16**, 578 (1930).

[4] Hendricks, S. B., *Zeits. Krist.* (In press).

of the fundamental translations in the brucite layer, which would cause the kaolin layer to curve." This statement would also be expected to hold for an iron analog of kaolinite.

Both the iron-bearing minerals, cronstedite and faratsihite, have been considered as related to the kaolins. The following work is a further study of these minerals, the former of which is particularly interesting because of the continuous scattering of x-radiation from some crystal zones.

The Crystal Structure of Cronstedite

Cronstedite is usually classified as a chlorite and is variously described as monoclinic or trigonal pyramidal. Commonly developed forms are: {00.1}, {10.2}, {20.1}, and {30.1}, referred to hexagonal axes.[5] Gossner[6] has studied the crystal structure of cronstedite from Kisbanya, Hungary. The analysis and succession of atomic layers parallel to the perfect cleavage, according to Gossner, are:

SiO_2	16.42%	2.00 mols.	Succession of layers
Al_2O_3	0.90	0.06	6, O
Fe_2O_3	29.72	1.36	2, Si and 2, Fe‴
FeO	41.86	4.26	4, O and 2, OH
CaO	1.32	0.17	2, Fe‴ and 4, Fe″
H_2O	10.17	4.12	6, OH

The structure as given by Gossner is isomorphous with point group C_s-m, and is related to that of kaolin. Dimensions of the unit of structure are $a_0=5.48$Å, $b_0=9.49$Å, $c_0=7.09$Å, and $\beta=90°$. Gossner's conclusion that iron is present in tetrahedral coordination is verified, but other details of the structure require revision.

Experimental Results

Work was greatly facilitated by use of splendid crystals of cronstedite from the Roebling and Canfield collections made available by Dr. W. F. Foshag of the U. S. National Museum. These included a specimen, number R 7807, from Kisbanya, that was closely similar to the one described by Gossner. Crystals more suited for x-ray work were obtained from specimen R 4550, Kuttenberg, Bohemia, and C 3733, Cornwall. The former contained thin trigonal plates similar to those from Kisbanya, but much less distorted. Crystals from the Cornwall specimen were elongated parallel to the c axis and were suitable for photographs taken with rotation about that axis.

Laue photographs taken with the x-ray beam normal to {00.1} of crystals from Kuttenberg had the symmetry of the point group $D_{3d}-\bar{3}m$.

[5] Dana, E. S., *A System of Mineralogy,* New York 656 (**1914**).
[6] Gossner, B., *Centralblatt. Mineral.,* 195 (**1935A**).

A number of continuous radial streaks were present in addition to a pattern of intense normal reflections. Gossner has stated that a Laue photograph of a crystal from Kisbanya showed monoclinic symmetry, but was badly distorted. Crystals from specimen R 7807 gave photographs that were too distorted to permit determination of symmetry.

Rotating crystal and equatorial and layer line x-ray goniometer photographs of cronstedite were taken with FeK and MoK radiation with rotation about the c axis and the normals to $\{10.0\}$ and $\{01.0\}$. All the photographs agreed with apparent trigonal symmetry, the $\{10.0\}$ normals being equivalent. Lattice dimensions obtained from these photographs and from measurements of the cleavage spacing against β Al_2O_3 as reference are:

$$a_0 = 5.48\text{Å} \qquad c_0 = 21.25\text{Å}$$

The corresponding monoclinic or orthohexagonal unit of structure has $a_0 = 5.48\text{Å}$, $b_0 = 9.49\text{Å}$, $c_0 = 21.25\text{Å}$, and $\beta = 90°$, c_0 being three times as great as Gossner's value.

Structure Determination

The Kisbanya material according to Gossner's analysis corresponds rather closely to

$$(Fe''', Al''')_{.84}(Fe'', Ca'')_{4.43}(Fe_2'''Si_2'''')O_{6.4}(OH)_{11.6}$$

the amount of water being somewhat high. This can be idealized for structural purposes as

$$(Fe''', Fe'', Al''', Ca'')_{4-6}(Fe_2'''Si_2'''')O_8(OH)_{10}$$

which is comparable with the composition of a layer in the unit of structure of a kaolin mineral, $Al_4Si_4O_{10}(OH)_8$, and a hypothetical kaolin mineral, $Fe_6Si_4O_{10}(OH)_8$. Moreover, the calculated molecular weight in a layer of the monoclinic unit of structure is 740, which, in view of the complexity of the mineral, is in agreement with 770, the value determined from the density (3.445, Gossner) and lattice dimensions.

The probable atomic arrangement in a layer of the cronstedite lattice is shown in Fig. 2 as a projection on (001) of Gossner's monoclinic unit of structure. Ferric iron is hardly to be considered as merely replacing silicon in tetrahedral coordination but only does so with accompanying separation of surrounding oxygen ions as is shown by the increase in a_0 and b_0. It is further improbable that silicon and iron randomly fill a set of positions with tetrahedral coordination; rather ferric ions fill just half of the positions. The hydroxyl content can be increased above

that of kaolin if each oxygen ion of a tetrahedron about ferric iron that is not shared between two tetrahedra is changed to hydroxyl in cronstedite.

Relative intensities for {00l} reflections were calculated for a kaolin-like structure of cronstedite with parameter values as shown in Fig. 2. These are compared with the observed values in Table 1 and it is perhaps evident that the agreement is very satisfactory.

TABLE 1. OBSERVED AND CALCULATED RELATIVE INTENSITIES FOR SOME REFLECTIONS FROM CRONSTEDITE—MoKα RADIATION, PSEUDO-HEXAGONAL INDICES (a_0=3.18Å, c_0=21.25Å) OF THE RHOMBOHEDRAL LATTICE

{00.l}	{30.l} {$\bar{3}$0.l}	{10.l}	{$\bar{2}$0.l}	{40.l}	{$\bar{1}$0.l}	{20.l}	{50.l}
l		l			l		
0	ms 80	2 vw 10	vw 8	vw 1	1 s 190	ms 110	vw 6
3 vs 820	mw 13	5 s 260	m 40	w 16	4 vs 510	s 130	w− 9
6 vs 670	mw 20	8 ms 60	vw 3	vw 2	7 vs 210	ms 60	vw 3
9 s 100	w+ 13	11 s 170	m 40	w 13	10 vs 350	s 100	w− 10
12 m 30	vw 3	14 s 150	mw 30	w 14	13 m 19	mw 9	
15 s 100	mw 30	17 mw 24	w 8	vw 4	16 m 60	mw+ 20	
18 m 60	w 13	20 m 50	w+ 18	vw 7	19 mw 22	mw 17	
21 m 45	mw− 17	23 a 0			22 vw 6	vw 2	
24 m 40	w 9	26 w 20			25 m 50	mw 27	
27 a 3		29 w 12			28 w 7	w 10	
30 w 15		32 a 9					
33 a 7		35 vw 12					
36 a 4							
39 w 14							

The diffraction patterns of cronstedite can be referred to an ortho-hexagonal unit of structure (O.H) having $a_0=5.48$Å, $b_0=\sqrt{3a_0}$, and $c_0=3\times 7.083=21.25$Å. Continuous scattering is present along lines connecting $\{h_a k_b l\}$, $k\neq n\times 3$, reflections. Thus on the photograph reproduced in Fig. 1 the {11l}, {15l}, and {17l} reflections are very weak (note Table V) and are connected within a group by continuous scattering. The {13l} and {19l} reflections are very intense and are not accompanied by general scattering.

Reflections for which the k index is a multiple of three, although varying in intensity, are very strong on the average. They can all be referred to a rhombohedral lattice having $a_0 = 3.16\text{Å}$ and $c_0 = 21.25\text{Å}$ as hexagonal

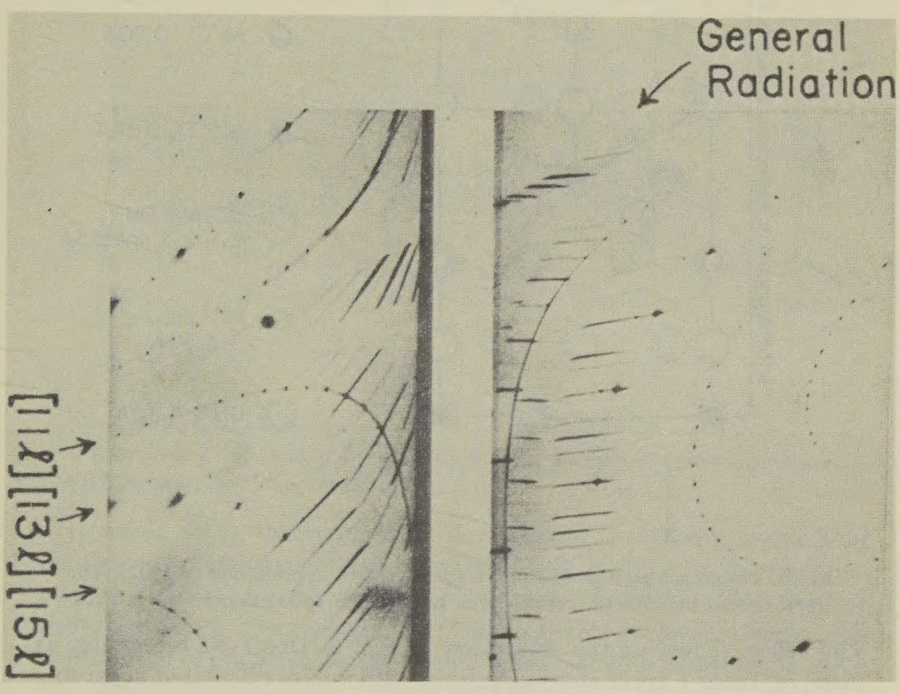

FIG. 1. Weissenberg x-ray goniometer photograph of the first layer line with rotation about the orthohexagonal a axis of cronstedite. Continuous streaks crossing lines connecting $\{h_a k_a l\}$ reflections are due to general radiation present with $\text{FeK}\alpha$.

translations (H). The transformations from orthohexagonal (O.H) to hexagonal (H) indices are

$$H = \frac{h-k}{2}(\text{O.H}) \quad L = l(\text{O.H}) \quad K = k(\text{O.H}).$$

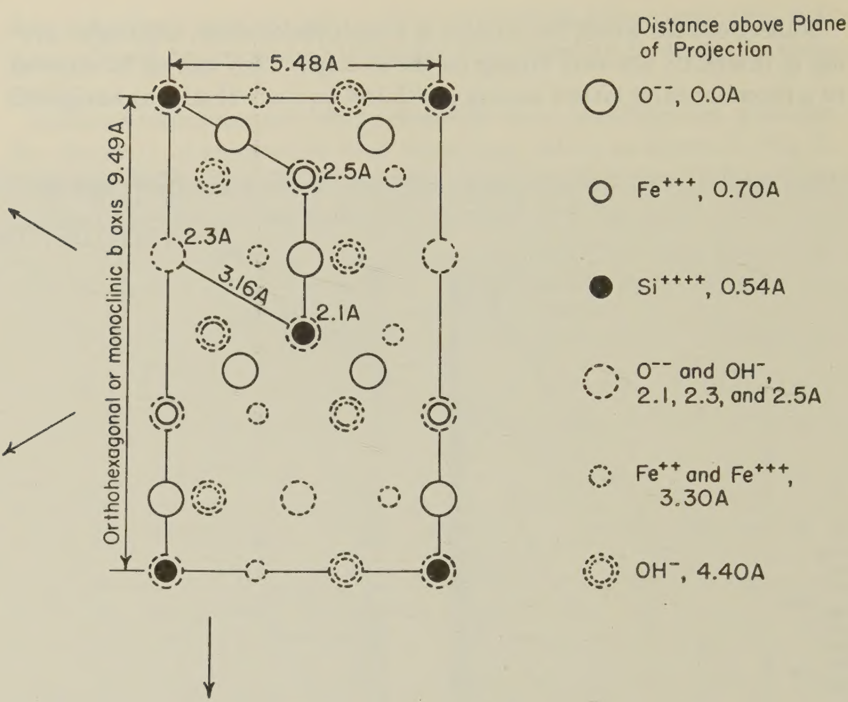

FIG. 2. The atomic arrangement in cronstedite shown in projection on {001}. The three arrows indicate the directions of the random translation in the lattice.

The cell is shown on the projection in Fig. 2. Observed intensities as listed in Table 1 are in close agreement with values calculated according to the following average arrangement given by space group C_3^4-R3:

Number	Atom	x	y	z
3	$\frac{Si+Fe}{3}$	0	0	.03
3	.9 Fe	0	0	.83
3	$\frac{O+2OH}{3}$	0	0	.11
3	OH	0	0	.54
9	$\frac{O}{6}$	$\frac{1}{2}$	0	0
9	$\frac{O}{6}$	0	$\frac{1}{2}$	0

Similarly good agreement between calculated and observed intensities was obtained for $\{13l\}$, $\{19l\}$, and $\{06l\}$ (O.H) reflections.

TABLE 2. OBSERVED INTENSITIES OF $\{0kl\}_{O.H.}$ REFLECTIONS OF CRONSTEDITE—FeKα RADIATION

l/k	0	2	4	6	8
0		w+	w		
1		−			
2		+			
3		+			
4		−	+		
5		+	−		
6		+	+		
7	over exposed	−	+	over exposed	present but very weak
8		+	−		
9		+	+		
10		−	+		
11		−	−		
12		−	+		
13		−	+		
14		+	+		
15		−	+		

NOTE: In order to bring out $\{0kl\}$ with k not a multiple of 6, it was necessary to strongly over expose $\{0kl\}$, $k=n\times 6$.

It would seem, therefore, that the structure is correctly represented by the averaged arrangement. Nevertheless it is still probable that the atomic positions in any one layer are those leading to the structure shown in Fig. 2. The averaged structure apparently results from a random shift of $nb_{O.H}1/3$ along the b axis of one layer with respect to another, the layers otherwise being stacked with trigonal symmetry. These random shifts correspond to the separation of the hydroxyl groups within a layer and are without effect on the relative positions of the hydroxyl groups in the top of one layer and the oxygen ions in the bottom of the adjacent layer.

Increased lattice dimensions as compared with the kaolin minerals which result from the presence of ferric iron with tetrahedral coordination permits almost complete filling of octahedral coordination positions by Fe^{+++}, Fe^{++}, Ca^{++}, Al^{+++}. Since practically all of these positions are filled, the coordination figures become more closely equivalent and the entire structure becomes more symmetrical. As a result of this regularity the layers can be stacked in a less ordered manner than is the case for nacrite where the individual layers are somewhat distorted from the ideal arrangement. The same condition is beautifully shown in the polymorphism of the micas.[7]

[7] Hendricks, S. B., *Nature*, **143,** 800 (1939).

Diffuse scattering in cronstedite is restricted to those zones having constant h and k indices with variable l index, the k index not being a multiple of three. There is thus an immediate and necessary connection with the random structure that leads to the pseudo unit of structure. Unordered translations of various layers by $nb_0/3$ would not alter the atomic arrangements relative to planes having $k=n\times 3$. Such reflections, therefore, should be sharp as they are observed to be. In other zones, however, the indefinite sequence of layers would give an indefinite sequence of atomic positions which would necessarily lead to diffuse scattering in those zones. In a crude way this is equivalent to variation in the length of the c axis by integral multiples of a fundamental spacing.

While scattering along lines connecting $\{h_ak_al\}\, k_a\neq n\times 3$, reflections is continuous from cronstedite crystals, normal narrow reflections are present which, though very weak in comparison with $\{h_ak_al\}\, k=n\times 3$ reflections as a class, are more intense than the neighboring continuous scattering. Pseudo-rhombohedral indices cannot be assigned to these reflections. They indicate that the structure is not entirely random throughout but is regular in a very small fraction of a crystal.

Dickite,[8] pyrophyllite,[9] and talc[9] all have random structures similar in type to that of cronstedite but their crystals show little or none of the diffuse scattering along lines connecting $\{h_ak_al\}$, $k_a\neq n\times 3$, reflections. A partial explanation for this is that the several structures vary in randomness. Dickite, pyrophyllite and talc crystals probably have large regions of order in which the sequence of layers is that required by a monoclinic space group, separated by not necessarily more than a single layer with some other shift of $nb_0/3$. This type of structure would prevent calculation of intensities for $\{hkl\}\, k\neq n\times 3$ reflections, but would not lead to observable continuous scattering. As the number of random layers increases the intensity of the diffuse scattering should increase in the neighborhood of the normal reflections and should depend in intensity upon them. Cronstedite thus is an extreme case.

Similar diffuse scattering has been observed for micas, vermiculites, chlorites and stilpnomelanes in all of which random structures are developed to various extents.

The Nature of Faratsihite and Possible Iron Content of Kaolins

Lacroix,[10] who originally described faratsihite, considered it to be a kaolin in which part of the aluminum had been replaced by iron. The

[8] Hendricks, S. B., *Am. Mineral.*, **23**, 295 (1938).

[9] Hendricks, S. B., *Zeits. Krist.*, **76**, 211 (1930).

[10] Lacroix, M. A., *Bull. Soc. France de Mineral*, **37**, 231 (1914).

mineral has recently been studied by Gruner[11] and he has decided that it is strictly a nontronite; that is, a single layer in the lattice is formed of two $(Si_2O_5)_n^{--}$ sheets jointed by octahedral coordination about iron.

A number of x-ray and electron diffraction patterns were made from a sample of Lacroix's original faratsihite obtained from Dr. C. S. Ross of the U. S. Geological Survey. Ordinary x-ray powder diffraction data were essentially in agreement with those listed by Gruner. The important features of these data for present arguments are:

Spacing of reflection	Intensity
13.9Å	6
7.1	indistinct
4.87	0.5
4.44	3
3.54	2
1.519	2
1.481	2

Samples of faratsihite oriented by settling from suspensions in water and examined after the method of Bradley, Grim and Clark[12] showed greatly enhanced intensities of reflections for the 13.9, 7.1, 4.87, and 3.54Å lines. In particular the 4.87Å reflection in such photographs was more intense than the one at 4.44Å. It would seem, therefore, that these four lines must be $\{00l\}$ reflections of micaceous minerals. However, at least two minerals must be present since the lines do not form a regular sequence of orders.

Gruner really had considerable evidence that both faratsihite and nontronite from White Oak Creek near Bakersville, N. C., were mixtures of two minerals, one related to montmorillonite, the other to kaolinite. Thus the 13.9Å reflection of faratsihite disappeared upon heating to 300° while that at 7.1Å was unaffected. Moreover, the 1.481Å reflection is in the position required for one of the strongest lines of the kaolin pattern, and while it is present for the above two minerals, it is absent for a number of other nontronites that also failed to give reflections near 7.0Å. The reflection at 13+Å for these other nontronites was shifted to 9.5–10Å upon heating to 300°.

Differential heating curves as reproduced in Fig. 3 also clearly indicate that faratsihite is a mixture containing a large amount of a kaolin mineral. The essential feature of the nontronite curve shown by faratsihite is an absorption of heat, influenced by the relative humidity, in the region 150–200°. The prominent heat absorption near 600° is a characteristic feature of kaolins, accompanying their loss of water of composition.

[11] Gruner, J. W., *Am. Mineral.*, **20**, 475 (1935).

[12] Clark, G. L., Grim, R. E., and Bradley, W. F., *Zeits. Krist.*, 93, 307 (1938).

Absence of the same type of exothermic reaction in faratsihite, as shown by kaolinite above 900° and a kaolinite-nontronite mixture, is not unexpected, since the recrystallization of the amorphous Al_2O_3 is greatly influenced by other components of an intimate mixture. The small

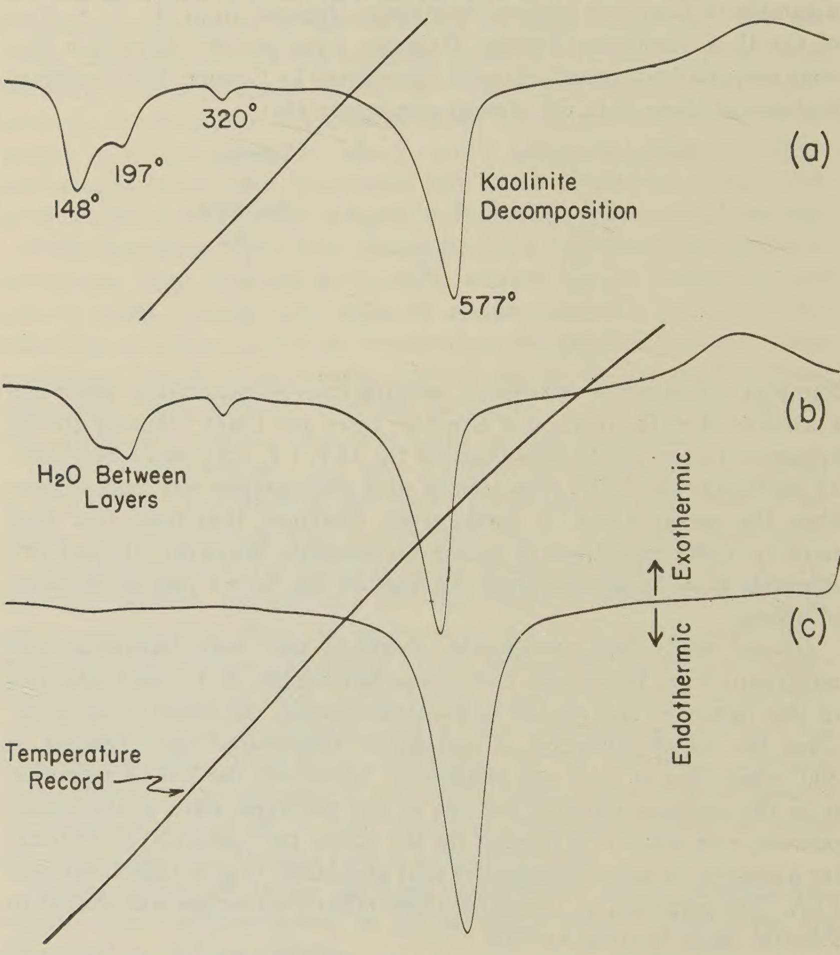

FIG. 3. Differential heating curves of (*a*) faratsihite, 75% relative humidity, sample weight 0.50 g. (*b*) faratsihite, 50% relative humidity, sample weight 0.44 g. (*c*) kaolinite, 50% relative humidity, sample weight 0.36 g.

endothermic reaction near 300° is in the region in which goethite loses water. However, treatment with nascent H_2S after the method of Truog and his associates, which was kindly carried out by Dr. L. T. Alexander of the Bureau of Plant Industry, indicated that no free oxides or hydrous oxides of iron were present.

Despite this confusing situation, it does not seem probable that the kaolin mineral in the mixture contains any appreciable amount of iron. The chief evidence for this is that diffraction patterns of faratsihite have reflections in the expected positions for ordinary kaolinite.

Raoul's Analysis of Lacroix's Material[10]:

SiO_2	41.60%	0.692 mols.
Al_2O_3	22.68	.222
Fe_2O_3	15.22	.095
FeO	0.54	.007
MgO	0.11	.003
CaO	0.60	.011
Na_2O	0.16	.002
K_2O	0.22	.002
TiO_2	0.13	
P_2O_5	0.21	
H_2O at 150°	5.71	
H_2O at red heat	13.02	

The amount of water lost above 150° considerably exceeds that expected for nontronite and approaches that required for kaolin. If the $Al_2O_3:SiO_2$ ratio of the kaolin present is assumed to be 1.7:1 then the ratio $(Fe_2O_3 + \frac{2}{3}FeO):SiO_2$ is 3.14:1, which is well within the possible range of nontronite. The sample would contain about 60 per cent kaolin and this is in approximate agreement with the heating curves which suggest 50 per cent.

Since the kaolin and nontronite present in the mineral faratsihite apparently grew together from solution, it would seem that conditions might have been favorable for formation of an iron-bearing kaolin. Its absence thus would suggest that such a compound is quite unstable as also is indicated by the particular structure of cronstedite.

Summary

The mineral cronstedite $(2FeO \cdot Fe_2O_3 \cdot SiO_2 \cdot 2H_2O)$ is formed of kaolin-like layers containing ferric ions with tetrahedral and octahedral coordination of surrounding oxygen and hydroxyl ions. These layers are superimposed with a random mixing of three possible structures which results in diffuse reflections along lines connecting $\{h_a k_a l\}$ reflections for which k_a is not a multiple of three. Although cronstedite is related to kaolinite $(Al_2O_3 \cdot SiO_2 \cdot 2H_2O)$ in structure it is improbable that the kaolinite lattice will accommodate appreciable amounts of iron in solid solution. The mineral faratsihite is shown to be a mixture of nontronite and an ordinary kaolin mineral.

ASSOCIATION OF HARMOTOME AND BARIUM FELDSPAR AT GLEN RIDDLE, PENNSYLVANIA

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Abstract

Small crystals of harmotome with a cruciform penetration twinning habit were found on the joint surfaces of a dike of anorthositic gabbro flanked by serpentine at Glen Riddle, Delaware County, Pennsylvania.

The dike is composed of two distinct rock types. (1) A hard gabbroic border phase consisting chiefly of sodic labradorite and 25 per cent biotite. (2) A crumbly central portion consisting chiefly of sodic oligoclase and 10 per cent biotite. Irregularly spaced crush zones parallel to the vertical foliation in the border rock contain corundum, hyalophane (Cn_{15}) and andesine (An_{40}). Similar zones occurring chiefly along the margins of the central portion contain hyalophane (Cn_{10}) and oligoclase (An_{15-20}).

The feldspars in the border rock show strain effects, and the crush zones contained therein show evidence of hydrothermal reaction and replacement such as hyalophane-plagioclase antiperthites. Similar zones in the central portion show little evidence of strain and contain patchy aggregates of hyalophane and albite-oligoclase. The most alkaline zones in the central portion reveal no evidence of strain. In these zones, sparsely distributed grains of hyalophane with indented and frayed borders are surrounded by albite-oligoclase.

The contemporaneous deposition of the hyalophane and corundum and the later deposition of the harmotome is considered to be the result of (1) deformation of a three foot wide dike of anorthositic gabbro, emplaced in pyroxenite, by forces accompanying the intrusion of a hornblende granite close by, and (2) infiltration of hydrothermal solutions (or superheated vapors) rich in potash, barium and soda, which emanated from the granite at an advanced stage of cooling, and which initiated a series of reactions along favorable channels within the dike rock, resulting in the deposition of the mineral suites described.

The hyalophane-andesine antiperthites with corundum are considered to have been formed at relatively high temperatures; the hyalophane-albite-oligoclase patch perthites without corundum, at lower temperatures; and the association of albite-oligoclase with indented grains of hyalophane, as representing partial resorption of previously formed hyalophane by late soda-rich solutions which deposited the resorbed potash and barium as harmotome in the joints and fissures of the dike rock.

The value of 2V for harmotome of 79°50′ (described in Section 2) is at variance with figures given in standard texts, and suggests possible oversight on the part of workers to note the effect of ordinary heat of grinding on the optical properties of the mineral.

Introduction

The barium zeolite harmotome has been reported from so few American localities[1] that its occurrence in a gabbroic dike at Glen Riddle, Pa.,

[1] Manhattan Island, and near Port Arthur, Lake Superior: Ford, W. E., *Dana's Textbook of Mineralogy*, 2d ed., 551 (**1922**). Sing Sing, N. Y.: Specimen in the collection of Mr. Hugo Bilgrim, Philadelphia, Pa. Thunder Bay, Canada: Doelter, C., *Handbuch der Mineral Chemie*, Band **II**, 404 (1919).

in close association with barium feldspar and corundum has been considered of sufficient importance to merit a detailed description. The dike is exposed in a railroad cut along the Octoraro Branch of the Pennsylvania Railroad, just west of the Glen Riddle station, about 17 miles southwest of Philadelphia. The harmotome crystals are scattered over the joint surfaces of the dike rock, while the barium feldspars occur chiefly as separate grains, or as hyalophane-plagioclase antiperthites, along crush zones or solution cracks in the rock matrix. Two distinct types of hyalophane are present: one whose composition is $Or_{90}Cn_{10}$, and a second whose composition is $Or_{85}Cn_{15}$. So far as is known, no occurrence of the second type has been reported thus far in the literature.

The first part of this paper describes a petrographic study of important variations in the dike rock and of closely related major formations in the vicinity. From the results obtained, an interpretation of the probable paragenetic sequence is offered. The second part describes the crystallography, physical properties and chemical analysis of the harmotome found at Glen Riddle, together with physical changes induced by heating the mineral. The method used for the identification of the barium feldspar is also described.

The writer has made a joint study of the Glen Riddle dike with Mr. W. Harold Tomlinson, who has described the paragenesis of the corundum in a separate paper.[2]

I

Geology of the Glen Riddle Area

The geology of the immediate vicinity is shown on the map (Fig. 1). The black wavy line marked *H* is the dike where the harmotome was found. It is flanked on either side by serpentine (an altered pyroxenite). A slightly gneissic hornblende granite lies in contact with the serpentine about eight feet east of the dike, and for the next 200 feet eastward granite and serpentine alternate with one another in close succession. To the west of the dike the rock exposed in the railroad cut is dominantly serpentine with a few narrow bands of granite exposed at broadly spaced intervals. Three hundred feet northwestward, across the strike, a body of metamorphosed gabbro about one hundred feet wide is exposed in a creek valley. Good exposures of meta-gabbro may be found on all sides of the mapped area. All the formations including the dike have a rough schistosity or gneissic structure which trends in a general NE-SW direction and dips steeply to the SE. The gabbro and granite have been

[2] Tomlinson, W. Harold, Corundum in a dike at Glen Riddle, Pa.: *Am. Mineral.*, **24**, 339–43 (1939).

considered to be intrusive into the serpentine,[3] and the latter into the Wissahickon gneiss.[4]

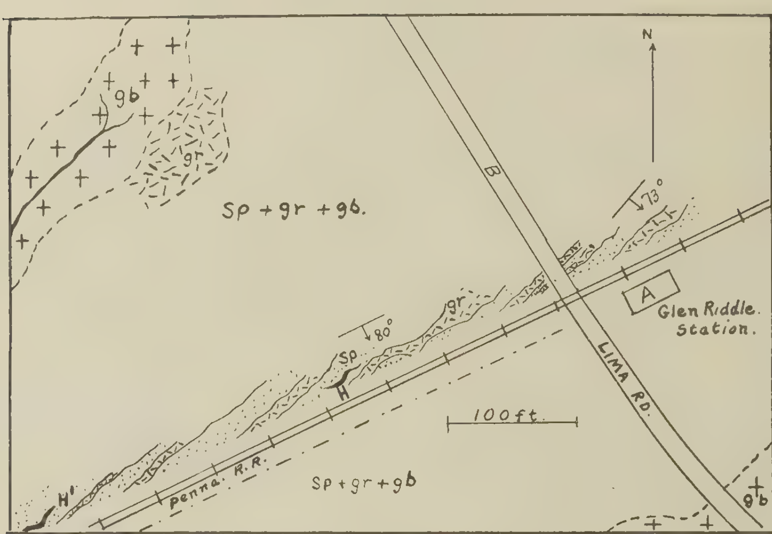


FIG. 1. Map of Formations at Glen Riddle, Pa.

H. Gabbroic dike where harmotome, corundum and barium feldspars were found.

H.′ Gabbroic dike similar to H containing barium feldspar, and corundum, but without harmotome, and without the crumbly central portion peculiar to H.

Serpentine (Meta-pyroxenite)

Granite

Metagabbro

Serpentine, granite, metagabbro (contacts poorly defined)

A. Glen Riddle station. Pennsylvania Railroad, Octoraro Branch.
B. Lima road. Penna. route 452.

[3] Watson, E. H., *Report of the Fifth Annual Field Conference of Pennsylvania Geologists*, 32 (1935).

[4] Bascom F., *U. S. Geol. Survey, Geol. Atlas, Philadelphia Folio* (No. 162).

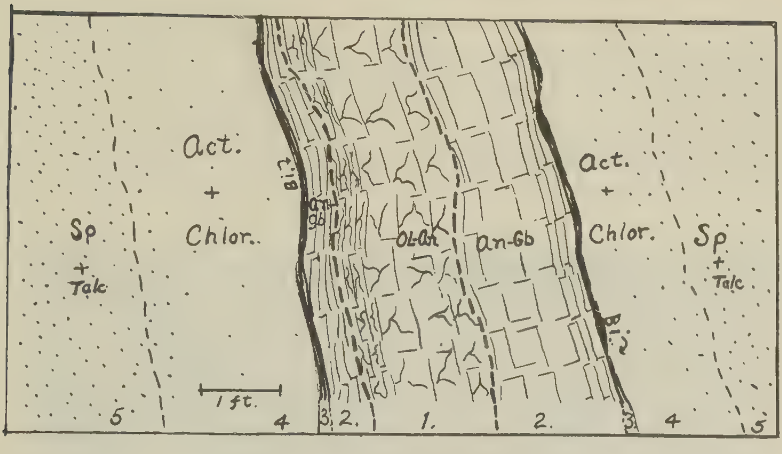


Fig. 2. Cross Section of Dike.

1. Friable central portion. Ol–An—Oligoclase-anorthosite. (See types E and F in text.)
2. Hard border rock. An-Gb—Anorthositic gabbro. (Types C and D.)
3. Thin biotite or vermiculite contact layer.
4. Actinolite and chlorite zone of "contact" minerals.
5. Serpentine wall rock containing occasional talc and tremolite.

Scheme of marking zones 1 and 2.

Vertical lines, foliation.
Horizontal lines, jointing (prominent direction).
Irregular wavy lines in zone 1, random cracks or veins.
Broken vertical lines, transitional zones between 1 and 2.

Harmotome Crystals

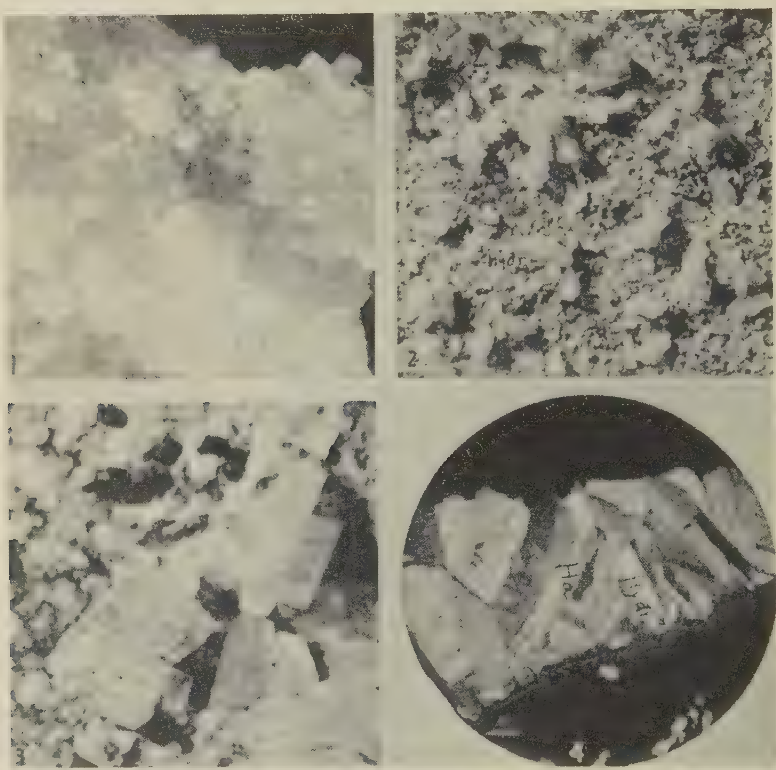
The harmotome crystals found at Glen Riddle are well developed cruciform penetration twins measuring from 3–5 mm. in length and 2–3 mm. in width. They are cream colored or white to crystal clear with a pearly to glassy luster. (Figs. 1–3, Pl. 1.) They are generally found resting on a semi-transparent film of crystalline hydrargillite (Figs. 2 and 4, Pl. 1). Separate crystals or thin drusy coatings are rather sparsely distributed along the joints of the dike which lie in a nearly vertical position against serpentine (altered pyroxenite) wall rock.

Structure and General Composition of the Dike

The structure of the dike, a cross section of which is shown in Fig. 2, suggests that it has undergone considerable deformation as well as later injection since the time of its original emplacement. The foliation is well marked by thin layers of biotite or vermiculite, which are closely spaced at the borders (one half inch), and more broadly spaced toward the center of the dike (4–6 inches). Small-scale horizontal faulting has broken the

rock into a number of loosely fitting blocks of various sizes. A narrow zone of biotite or vermiculite sharply delimits the greyish black border rock of the dike from the bluish green highly altered pyroxenite on either side. The blue green color is caused by a mixture of actinolite and chlorite which grades off into a mixture of serpentine and talc with occasional tremolite.

PLATE 1



Glen Riddle Harmotome.

1. Harmotome crystals showing cruciform penetration twins. ×4 diam.
2. Harmotome crystals resting on hydrargillite (Hydr.). ×4 diam.
3. Same as 2. Mag. about 8 diam.
4. Thin section of harmotome (Ha) resting on hydrargillite (Hydr.) ×nicols. Mag. about 20 diam.

Within the dike itself the rock immediately adjacent to the biotite contact is extremely hard. In composition and texture it resembles most closely the local gabbro, although slightly more anorthositic. It consists of approximately 75 per cent of felsic minerals, chiefly sodic

labradorite, and 25 per cent biotite. The central portion of the dike (bounded by the two heavy broken lines in Fig. 2) is light grey in color and crumbles easily. It consists of 90 per cent of felsic minerals, chiefly albite-oligoclase and hyalophane, and 10 per cent biotite. The broken lines (Fig. 2) represent a 2–3 inch wide transitional zone between the two more extreme types. These zones are rich in corundum (11 per cent), hyalophane and andesine.

The short horizontal lines in Fig. 2 represent cross joints in which the harmotome was found. The parallel vertical lines represent directions of easy cleavage along foliation planes. The random lines in the central portion represent countless cracks which have apparently served as channels for the flow of solutions through this part of the dike.

Lense-like masses in the central portion.

Relatively hard lense-like masses appear along the western border of the crumbly central portion of the dike. They are from 3–5 inches in length and possess a different composition from the soft crumbly rock which partly or completely surrounds them. They are of two types. The first type consists chiefly of hornblende and oligoclase. The composition of the second type is identical with that of the transitional zones,—*i.e.*, rich in corundum, andesine and hyalophane. Their probable bearing on the history of the dike will be discussed in connection with the paragenetic sequence.

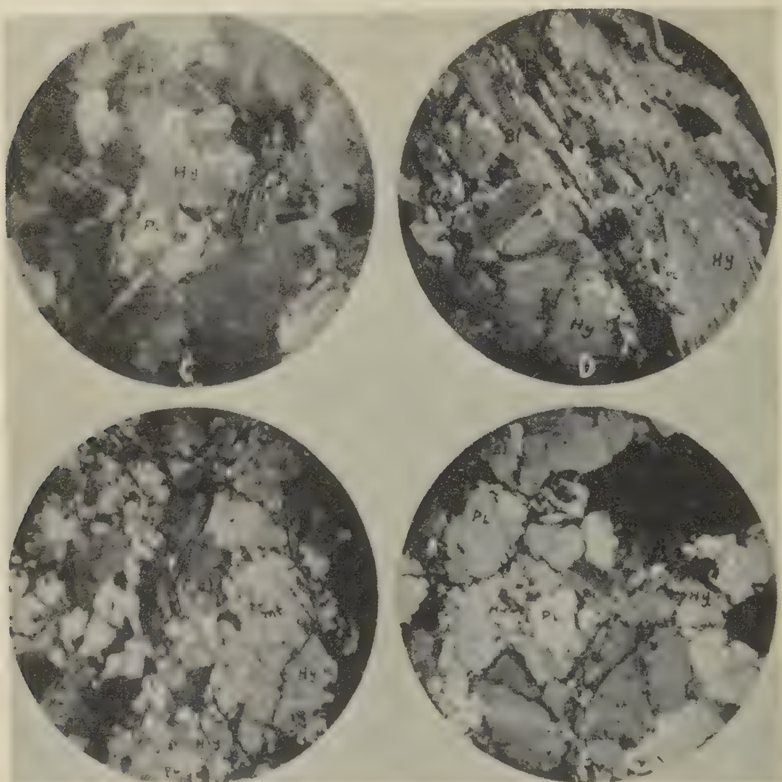
Petrographic Analysis

Under the microscope irregularly spaced crush zones about 1–2 mm. wide appear parallel to the visible foliation in almost every phase of the dike rock. They are spaced from 4 mm. to 75 mm. apart. It is along such crush zones—sharply defined in the hard border rock, and partly obliterated in the central portion—that the potash-barium feldspars and corundum are found. Except for its presence in the lense-like masses already mentioned, the corundum is confined exclusively to the narrow crush zones in the border rock and to the transitional zones between the border and center. The minerals in the crush zones make up about 15 per cent of the bulk composition of the dike rock.

Photomicrograph *C* (Pl. 2) represents a typical section of the border rock; *D*, a typical crush zone in the border rock; *E*, a typical crush zone in the central portion,—lacking the sharp boundaries of *D*, and most abundant along the outer margins; and *F*, a typical section of the rock which makes up the greater percentage of the central portion of the dike. All photographs were taken under the same magnification in order to illustrate relative differences in grain size. Type *C* (with alternate

zones *D*) to *E* and *F* represent a typical cross section traverse from the border to the center of the dike.

PLATE 2



Important Rock Types in Glen Riddle Dike where Harmotome Was Found.

C and *D*, Border Phase. *E* and *F*, Central Phase.

X Nicols. Mag. about 24 diam.

Pl—Plagioclase. Hy—Hyalophane. Bi—Biotite. C—Corundum. mt—montmorillonite.

C. Dominant type in border phase. Single grain of antiperthite in center of field composed of plagioclase (An_{40}) and hyalophane (Cn_{20}).

D. Mylonitized zones in border phase. Two large grains of antiperthite in lower half of field. Separate grains of hyalophane in center. Small seed-like xls. of high relief are corundum.

E. Mylonitized zones in central phase, top left, with patch-work mosaic at bottom center of field. Large grain of antiperthite, lower right with part of plagioclase altered to montmorillonite (mt).

F. Dominant type in central phase. Shows a few small grains of hyalophane in center of field.

TABLE 1. MINERALOGIC COMPOSITION OF GLEN RIDDLE METAGABBRO AND VARIOUS PHASES OF THE DIKE ROCK

			Border Phase		Central Phase		Av. Grain Size*	
	A	B	C	D	E	F	mm.×mm.	
Hornblende	51	20					1.0	.80
Biotite		5	26	31	7	10	.33	.14
Quartz	10.5							
Plagioclase								
An_{60}	36		60	5				
An_{40}			4	30			.77	.58
An_{20}		49			24	20		
An_{15}					8	36		
Hyalophane								
Cn_{15}			2	9			.20	.12
Cn_{10}		16			26	7		
Antiperthite								
$An_{40}+Cn_{15}$			5	12–19			1.00	.81
$An_{20}+Cn_{10}$		8			11	5		
Corundum			1	4–11			.04	.02
Accessories								
Apatite	1.5						.22	.16
Zircon								
Titanite	*m* 1.0	2	2	2	1	2	.02	.01
Secondary Montmorillonite, clay, etc.					23	20	n.d.	
Av. grain size in mm. *l.***	Same	.89	.54	.37	.55	.63		
w.	as C.	.67	.38	.20	.40	.43		

A. Local metagabbro. *m* = magnetite—present in A only.
B. Lense-like inclusions found in type E of central phase.
C. Dominant type in border phase.
D. Mylonitized zones in border phase.
E. Mylonitized zones in central phase bordered by patch-work mosaic.
F. Dominant type in central phase.

* Average dimensions of each mineral for all phases in which it is present.

** Average grain size of each phase excluding corundum, accessories and secondary minerals. 18 to 35 grains were measured on each slide. *l.* = length. *w.* = width.

Summary

A summary of the study of the photomicrographs and of the data presented in Table 1 and Fig. 3 reveals that as we pass from the border to the center of the dike the plagioclase becomes more sodic, the hyalophane becomes more potassic, grain size increases, evidence of strain diminishes, crush zones (reaction zones) become broader, antiperthites decrease, separate grains of hyalophane increase in number and finally decrease notably in the most sodic central part of the dike.

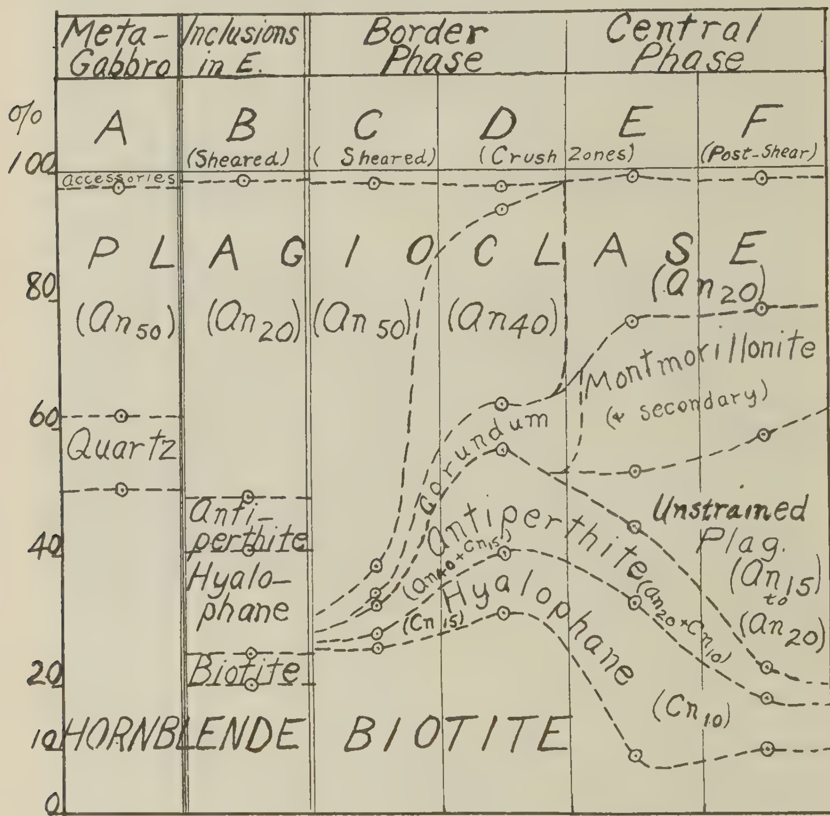


FIG. 3. Variation Diagram. Constructed from Analyses in Table 1.

Table 1 represents an analysis of the mineral content and grain size of the local meta-gabbro and outstanding types found in the Glen Riddle dike. Types *C*, *D*, *E* and *F* correspond to photomicrographs (Pl. 2) bearing the same letters and represent a cross section from the border (*C* and *D*) to the center (*E* and *F*). In the variation diagram (Fig. 3) columns *C*, *D*, *E* and *F* show the progressive appearance and disappearance of the components of each phase from the basic border to the more alkaline center of the dike.

Mineral percentages were determined by the Rosiwal method with the Wentworth integrating stage. Five to seven traverses were made on each slide. *D* and *F* represent the average of two slides for each type. Count was made on one slide only for each of the remaining types. Additional slides and crushed samples were examined in order to insure a reasonably dependable degree of accuracy. The plagioclase feldspars were determined by measurement of refractive indices on {010} and {001} cleavage plates, and by referring to curves in standard textbooks. The method for the determination of the hyalophane is described in section 2 of this paper.

Paragenetic Sequence of Barium Minerals

Since the relations obtaining in the dike are not nearly so simple as might be gathered from the foregoing description, the interpretation which follows is presented in the form of a discussion rather than as a positive interpretation.

Source of the magma of the dike minerals.

1. The local gabbro is considered the most probable source magma of the dike as originally emplaced. This is suggested by the anorthositic composition of the hard border rock. The anorthite content of its labradorite is similar to that of the gabbro (45–50 per cent). Its content of mafic minerals (26 per cent) is much higher than that of the local granite (10 per cent). Accessory minerals also correspond to those present in the gabbro.
2. The local granite is the most probable source of the barium.
   (a) Hyalophane-plagioclase antiperthites similar to those in the dike rock were found in granite near Lenni Mills, Pa., about one half mile west of Glen Riddle.
   (b) Evidence of strain or shear in the dike rock wherever hyalophane-plagioclase antiperthites are found suggests that the original gabbroic rock of the dike was sheared by forces attending the intrusion of the granite magma, and that during or shortly after deformation highly mobile solutions emanated from the granite mass at an advanced stage of cooling, and soaked into the dike rock along microscopic cracks or crush zones.

Evidence of hydrothermal reaction from high to progressively lower temperatures, in the form of a reaction series.

(a) Chemical evidence. There is a progressive increase in the sodic content of the plagioclase from the border to the center of the dike. The anorthite content decreases from 50 to 15 per cent.

As the plagioclase becomes more sodic its associated hyalophane becomes more potassic. Where associated with labradorite and andesine the composition of the hyalophane is $Or_{85}Cn_{15}$; where associated with albite-oligoclase its composition is $Or_{90}Cn_{10}$.

(b) Physical evidence of progressively lower reaction temperatures from border to center.

1. Changes in texture of replaced rock and replacing minerals. In the resistant border rock where traces of the first attack of granitic volatiles are well preserved, the texture is dominantly antiperthitic. In the reaction zones which border the most sodic central portion, antiperthites have given way to an irregular patchy mosaic pattern of hyalophane and albite-oligoclase. (See Pl. 2, *D* and *E*.) Andersen[5] suggests that antiperthites indicate reaction at relatively high temperatures, whereas patch perthites are formed at relatively low temperatures. If his conclusions be applied here, hydrothermal reaction and replacement persisted down through a lower temperature in the central than in the more resistant border rock of the dike.
2. Formation temperature of the harmotome. The lowest temperature in the hyalophane-harmotome sequence would be represented by the appearance of the barium zeolite. At this stage the rock would have cooled sufficiently to form contraction joints into which the excess liquid would find its way. The formation temperature of the harmotome, as suggested by its behavior on heating (Section 2 of this paper) may have been as low as 180–200°C.

Transition from hyalophane to harmotome.

While the record of changing composition and temperature is rather clearly preserved in the case of the hyalophane and plagioclase feldspars, the intermediate steps between the lowest temperature of formation of hyalophane and that of harmotome are not so clearly marked. The explanation which appears to be most in keeping with the evidence at hand, is that after the deposition of the last formed hyalophane the granitic solutions became progressively richer in soda, and by a process of metasomatic replacement resorbed an amount of hyalophane equivalent to the relatively small amount of harmotome which is now sparsely distributed along the joints of the dike rock. This resorption appears to have taken place along the central channels of the dike where the rock (type *F*) most nearly resembling the granite is found.

An alternative explanation would be that as the solutions cooled down they became progressively richer in barium, potash and water, and at the proper conditions of equilibrium produced the crystals of harmotome.

[5] Andersen, O., The genesis of some types of feldspar from granite pegmatites: *Norsk geol. tidsskr.*, B. X. h. 1–2, 151–204 (1928).

The great excess of soda over potash in the latest formed phases of the dike, together with certain considerations enumerated below, mitigates against this relatively simple explanation.

Metasomatic replacement of central portion of dike by late soda-rich granitic injections.

(a) Chemical evidence. In the central portion of the dike (type *F*) the ratio of soda to potash feldspar is as 8:1, whereas in the local granite it is as 1:3.

(b) Evidence of prolonged and intense soaking in the central portion. Reaction between granitic volatiles and original dike minerals is not confined to narrow crush zones, as in the hard border rock, but has spread out in a haphazard fashion, forming zones of extended reaction. (Compare Pl. 2 Figs. *D* and *E*.) The lense-like masses, some containing hornblende as the mafic mineral, and others rich in corundum, appear to be remnants of more extensive zones which were either present in the original gabbroic dike or were formed at an early stage of the granitic sequence.

1. The hornblendic masses are believed to be a part of the original gabbro dike. Their content of hornblende is equivalent to the biotite content of the border rock. Like the border rock they also contain antiperthites which show distinct evidence of strain. This should place them in the pre-granitic gabbro sequence.
2. The corundum-rich masses. Since corundum is always associated with hyalophane, the corundum-rich masses must belong to the early high temperature granitic sequence best preserved in the hard border rock.

(c) Absence of strain in the central portion. Marked evidence of strain in the hard border rock and in the hornblendic masses, such as strain shadows, curved twin lamellae and granulation, is only partially present in the reaction zones bordering the central portion; and disappears almost completely in the most sodic central zone of the dike. In the latter the twin lamellae of the feldspars are perfectly straight (see Pl. 2, type *F*). Almost complete absence of antiperthites, which are generally associated with stress phenomena in the dike, suggests a late period of formation for the most sodic central portion of the dike.

(d) Attack of minerals formed early in sequence by later solutions.

1. Borders of hyalophane grains are frayed and indented (see Pl. 2, type *F*).
2. Crosscutting tendency of invading minerals. Hyalophane intergrowths crosscut the cleavage traces of the plagioclase in the

hard border rock. In the central portion (type E) intergrowths of albite-oligoclase crosscut the cleavage traces of hyalophane.

Summary of Evidence of Replacement in Central Portion of Dike by Late Soda-Rich Solutions

From the evidence given above it appears that the potash-barium feldspars kept forming for some time after the relaxation of stress, but that the solutions finally became impoverished in barium and potash, and richer in soda and water. Increase in water may have facilitated diffusion along favorable passages and appears to have effected local replacement of some of the earlier reaction products. Some of the hyalophane appears to have been resorbed to form harmotome, while some of the corundum may have been resorbed to form hydrargillite, thus explaining the close association of harmotome and hydrargillite on the joints of the dike rock. The presence of 1.2 per cent of soda in the harmotome also suggests that soda was present in the solutions which deposited the barium zeolite.

If interpreted correctly, this evidence of replacement by soda-rich solutions confirms the work of Noll,[6] whose study on artificial hydrous silicate melts indicates that soda-rich solutions promote the formation of zeolites, whereas potash-rich solutions aid in the formation of such minerals as sericite. He also finds that an excess of alumina in water rich melts will produce corundum between 400–600°C. and böhmite (hydrargillite) at 300°C. or less.

The combined evidence of reaction and replacement in the dike rock as a whole is also in accord with the reaction principle of Bowen, and with the views of Butler, who extends the reaction series to include deuteric and hydrothermal phases. The results also confirm the views held by Schaller and Andersen, who investigated hydrothermal replacement phenomena in lithium and microcline pegmatites. Although the chief difference lies in the more basic nature of the replaced dike minerals at Glen Riddle, and the chemical nature of the granitic exudates which replaced them, the actual processes involved are essentially the same.[7]

Summary of Paragenetic Sequence of Glen Riddle Dike Minerals

A. Emplacement of a dike of anorthositic gabbro in pyroxenite at near

[6] Noll, W., Über die Bildungsbedingungen von Kaolin, Montmorillonit, Sericit und Analcim: *Min. pet. Mitt., Band* **46** Heft 3–4, 221–224 (1936).

[7] Bowen, N. L., The reaction principle in petrogenesis: *Jour. Geology*, **30**, 177–198 (1922).

Butler, B. S., Influence of replaced rock on replacement minerals associated with ore deposits: *Econ. Geology*, **27**, 1–24 (1932).

Schaller, W. T., The genesis of lithium pegmatites: *Am. Jour. Sci.*, **10**, 269–279 (1925).

Andersen, O., *Loc. cit.*

magmatic temperatures. This sequence is now represented by (1) the hard border rock, and (2) by the hornblendic masses which may have once extended as thin bands up the center of the dike.

B. Structural deformation of dike rock attended by the introduction of late-stage granitic exudates rich in potash, barium and soda. Reaction of solutions (or superheated vapors) with original dike minerals to form:

1. An early relatively high temperature stress sequence consisting of hyalophane (Cn_{15}), andesine (An_{40}) and corundum in the border rock, and of hyalophane (Cn_{10}) and oligoclase (An_{20}), without corundum, in the portions represented by the hornblendic inclusions. The temperature range was probably from 500–600°C. There were relatively equal amounts of soda, potash and barium in the invading solutions.
2. Low stress intermediate temperature sequence consisting of relatively large grains of hyalophane (Cn_{10}) and albite-oligoclase (An_{15}). This stage represented maximum potash and barium content in solutions with increase in water vapors. This was followed by:
   (a) Increase in soda content of solutions and local replacement of previously formed hyalophane by albite-oligoclase in the center of the dike.
3. Low temperature zeolitic sequence composed of harmotome and hydrargillite. Solutions rich in water resorbed potash, soda, barium and alumina. Temperatures approximately 200°C.

The change in the celsian content of the hyalophane from 10 per cent where associated with albite-oligoclase (An_{15}) to 15 per cent where associated with andesine (An_{40}) may be the manifestation of a possible established equilibrium. In his work on myrmekites Becke[8] found that the amount of quartz formed during the replacement of orthoclase by plagioclase increased in direct proportion to the increase in lime content of the invading feldspar. In the Glen Riddle occurrence the barium content of the hyalophane intergrowth appears to be controlled by the lime content of the invaded plagioclase. It is not known whether the lime or the alumina in the plagioclase is the actual controlling factor which determines how much barium will appear in the hyalophane, but since both calcium and barium belong to the alkaline earth group it is quite probable that where stoichiometric relations are involved these elements are interdependent. Because of close correspondence in the size of their atomic radii, however, the barium is able to replace potassium in the lattice

[8] Becke, F., Über Myrmekit: *Min. pet. Mitt.*, **27**, 377–390 (1908).

structure, and thus form hyalophane. It is worthy of note that hyalophane which occurs in limestone districts has a still higher barium content than any of the types found at Glen Riddle. Hyalophane reported from Franklin, N. J., has a celsian content of approximately 25 per cent.[9] Since the actual processes involved in the formation of simplectites are not very well understood, a mere mention has been made of this relationship without attempting to interpret its possible significance.

II

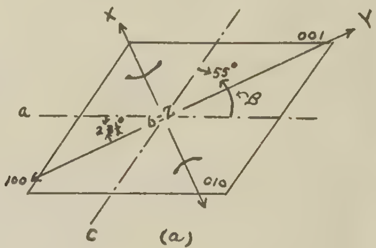
Crystallographic, Physical, and Chemical Properties of Harmotome from Glen Riddle, Delaware Co., Pennsylvania

Formula: $K_2Ba(Na_2Ca)O \cdot Al_2O_3 \cdot 5\ SiO_2 \cdot 5\ H_2O$. Monoclinic.

Forms Observed: *a*(100), *b*(010), *c*(001), *m*(110), see Fig. (5). *e*(011) present as twinning plane only.

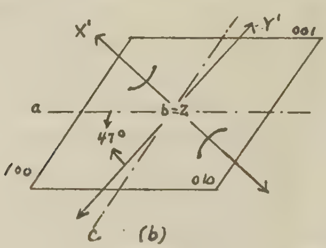
Twinning: On the base *c*(001) and on the clinodome *e*(011).

Habit: Cruciform penetration twins. In Fig. 5 the prism and the front pinacoid are about equally developed. In some crystals the prism face is absent; in others it has partially or completely replaced the front pinacoid.[10]



$Y \wedge a = 23\frac{1}{2}°$

Fig. 4. Optical Orientation of Harmotome below 100°C. on *b*(010).



$Y' \wedge a = 47°$.

Optical Orientation of Harmotome at 130°C. on *b*(010).

[9] Palache, Charles, The minerals of Franklin and Sterling Hill, Sussex County, N. J.: *U. S. Geol. Survey, Prof. Paper*, **180**, 60 (1935).

[10] The one crystal measured on a Goldschmidt two-circle goniometer had poor reflecting faces because of striations. The axial ratio, and the facial intercepts which are given above are in agreement with those given in *Goldschmidt's Winkeltabellen.*

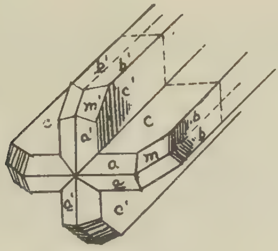


FIG. 5. Interpenetration Twins of Harmotome.

1. Showing forms observed.
2. Type of twinning.
3. Direction of striations observed on prism faces, $c(001)$ and $b(010)$.

CRYSTALLOGRAPHIC ANGLE: Beta$=55°$* (measured on thin section $\parallel$ to $b(010)$.
PHYSICAL PROPERTIES: Cleavages: $b(010)$ good, $c(001)$ fair. Gr.$=2.47$ H.$=4.5$.
OPTICAL PROPERTIES: Biaxial positive. $Z=b$. Axial Plane and $Bx_a \perp b(010)$. See Fig. 4(a). $\alpha=1.506$* $\beta=1.509$ $\gamma=1.514$ at 24°C.
Dispersion crossed; increases with heating.

$$2V_\gamma=79°50'†$$

* All readings marked * were made in yellow light with a Lifa Filter 395 D Na.
† Calculated from refractive indices. Values of $2V_\gamma$ obtained by measurements on separate grains on the Universal Stage were 78, 80, 81, and 82 degrees.

CHANGE IN OPTICAL PROPERTIES ON HEATING:[11] The extinction angle $Y \wedge a$, and the optic axial angle over Z vary with change in temperature as shown in Figs. 4 (a) and (b) and in Table 2 which also includes water loss on heating.

TABLE 2. OPTICAL CHANGES AND WATER LOSS ON HEATING OF HARMOTOME FROM DELAWARE CO.

Temp. °C.	$Y \wedge a$	$2V_\gamma$	Temp. °C.	Water Loss	Hours Heated
24	*$23\frac{1}{2}°$	79°50′	24	n.d.	
90–100	*24	*69°	105	4.9%	12
130	47	*32	140	6.8	15
180	55 ± 4	n.d.	190	10.3	4
**240	37–45	n.d.	240	11.6	3
**Red heat	37	n.d.	Red heat	14.8	1

** Extinction angles were read with difficulty above 180°C., owing to strain in the crystal fragments.

The changes in optical properties which attend the loss of water on heating are shown in Table 2. Similar changes in water loss have been noted previously by Fresenius (1879) and Stoklossa (1918). Optical

[11] Lack of agreement of optical data in various textbooks may be due to oversight on the part of workers to note the effect of heat. A thin section ground in the ordinary manner and mounted in balsam showed an optic angle of 32°.

changes have been observed by Des Cloizeaux (1868) and by Rinne (1890).[12] Approximately one third of the water is lost at 140°C. and an additional third at 190°. The optical angle $2V_\gamma$ becomes smaller, and the extinction angle $Y \wedge a$ becomes larger until its value coincides approximately with that of the crystallographic angle beta (55°). This coincidence takes place very close to 180°C. Further increase in temperature induces a reversal in the swing of the Y direction causing the extinction angle to become smaller again. Weigel has noted similar changes in the behavior of heulandite, another member of the desmine group, to which harmotome belongs.[13] The temperature at which coincidence and then reversal takes place is also in the neighborhood of 180°C. The point at which optical and crystallographic directions coincide is considered by Weigel to be the temperature at which the zeolite was originally formed.[14] Such a relationship in the case of harmotome could only be established by much more detailed work than appears in the above table. The value of 180°C. seems, however, to correspond fairly well with the low range of formation indicated by the petrographic study cited previously.

Chemical Analysis of Harmotome

As the number of available crystals was rather small, the analysis in Table 3, column A, was made on the purest fragments of the mineral found in the form of thin seams or crusts. After separation from impurities, mainly hydrargillite, the ground specimen was examined under the microscope. The amount of impurities was estimated to be not more than 1.5 to 2.0 per cent. Thin sections of harmotome oriented parallel to *b* (010) revealed tiny groups of acicular crystallites arranged in bundles. These were taken to be either thomsonite or natrolite.

Table 3. Analysis of Harmotome

	A	B	C	C′	E	F
SiO_2	45.51	47.1	46.15	.765	4.87	5
Al_2O_3	16.50	16.0	16.03	.157	1.00	1
MgO	0.27					
CaO	0.12		0.13	.003		
BaO	19.89	20.6	20.17	.131	1.10	1
K_2O	1.77	2.1	1.81	.019		
Na_2O	1.18		1.21	.020		
H_2O 110°C.	14.74	14.1	14.50	.806	5.13	5
Total	99.98	100.0	100.00			

[12] Doelter, *Handbuch der Mineral Chemie, Bd.* II, 404 and 406 (1919).

[13] *Doelter, Ibid.* 300 et seq.

[14] Angel and Scharizer, *Grundriss der Mineralparagenese*, 76–95 (1932).

A. Harmotome from Glen Riddle. Analyst A. E. Meier.

B. Theoretical composition of harmotome (*Dana's Textbook of Mineralogy*, 3rd ed., p. 551).

C. Analysis A calculated to 100 per cent after deducting an estimated 1 per cent of hydrargillite and an amount of hydrous magnesium silicate (white chlorite) equivalent to 0.27 per cent of MgO.

C′. Molecular proportions of C.

E. Molecular ratio of harmotome from Glen Riddle, computed from C. and C′.

F. Molecular ratio of theoretical harmotome, computed from B.

Analytical notes

The usual procedures recommended for silicate analysis by Washington and Hillebrand were followed. Barium was determined on a separate sample by the HF–H_2SO_4 method. The silica, alumina and lime precipitates were all purified and tested in order to correct for any occluded barium.

On a sample of harmotome weighing 0.3006 gms. the Al_2O_3 precipitate yielded 0.0017 gms. $BaSO_4$ (after double precipitation of Al_2O_3). HF residue from SiO_2 yielded 0.0015 gms. $BaSO_4$. Calcium oxalate ppt. was free from barium.

Magnesium ammonium phosphate brought down approximately 75 per cent of the barium. This precipitate was dissolved in HCl and the barium removed with H_2SO_4.

The filtrate from the main portion after all the other elements had been determined yielded 19.51 per cent BaO, against 19.89 per cent BaO obtained on a separate sample by the HF–H_2SO_4 method.

Identification of Barium Feldspar as Hyalophane

The presence of a number of mineral grains in type *D* (the mylonitized zones of the resistant border phase), whose highest interference color in thin section was a first order grey, and whose refractive indices were too high for orthoclase and too low for cordierite, suggested the possible presence of hyalophane in the dike rock. A chemical test made on the rock similar to photomicrograph *D* revealed a BaO content of 1.5 per cent. The refractive indices fell within the range of those given by Winchell[15] for hyalophane, and were halfway between those corresponding to a composition of $Or_{90}Cn_{10}$ and $Or_{80}Cn_{20}$. No composition was listed, however, which corresponded to the indices obtained.

In order that the exact composition of the unknown feldspar might be determined, an attempt was made to isolate the feldspar in question by means of heavy liquids. The first separation was made on unsized grains that passed through a 120 mesh sieve, and with potassium mercuric iodide as the heavy liquid. Gravity adjustments were made by means of a pycnometer and a standard analytical balance. After all the minerals heavier or lighter than sp. gr. 2.685 had been removed, there remained a mixture of almost equal parts of andesine (An_{40}) and an unknown feldspar. Portions of this mixture would either float or sink in a liquid of sp. gr. 2.685, the coarser particles tending to sink whereas the finer

[15] *Loc. cit.*, 360.

grains floated, or remained suspended. A second test was made on sized grains using bromoform, but no better separation was obtained. The fractions collected from each test were kept separate. Slides were made by immersing representative samples in liquids of suitable index, and the percentage of the unknown feldspar was determined by counting the grains. In the first test a number of grains (about 25 per cent of those counted) were measured, and the relative proportion of each feldspar present in the mixture computed by multiplying the number of grains counted on each slide by the average diameter. This procedure was not considered necessary in the second test since the grains had already been screened to size. No allowance was made for possible difference in vertical thickness of grains on the slides. Since both components of the mixture were practically inseparable in heavy liquids, per cent by volume and by weight were considered to be identical.

Table 4. Identification of Hyalophane by Gravity Separation and Chemical Analysis

	Test 1	Test 2
Liquid used	$KHgI_2$	Bromoform
Sp. gr. of liquid	2.685	2.686 at 20–25°C.
Screen sizes	through 120 mesh	{55% through 120 on 140 45% through 140 on 170}
Grains counted:		
Ba feldspar	243	689
Andesine (An_{40})	171	721
No. of grains measured:		
Ba feldspar	66	not measured
Andesine	60	not measured
Av. grain diameter:		
Ba feldspar	0.17 mm.	not measured
Andesine	0.20 mm.	not measured
Per cent present:		
Ba feldspar	55	47
Andesine	45	53
Wt. sample for anal.	0.1745 gs.	0.4012 gs.
Per cent BaO found:		
In mixture by analysis	3.76	3.08
In 100% Ba feldspar	6.46	6.55
In theoretical celsian	40.8	40.8
Per cent Cn in Ba feldspar	15.83	16.05
Per cent $Or_{85}Cn_{15}$ sol. in andesine (An_{40}) approx.		10*
Per cent celsian sol. in andesine (An_{40}) approx.		1.5
Per cent celsian remaining for free Ba feldspar		14.5
Approximate celsian content of Ba feldspar present in the border phase (type *C*) of dike		Cn_{15}

* This value was obtained by substituting hyalophane $Or_{85}Cn_{15}$ for orthoclase in the ternary diagram of Winchell, *loc. cit.*, 319.

Each of the two mixtures were analyzed separately for barium. The results are given in Tables 4 and 5. The computations are based on the assumption that all the barium found in the mixture is contained in the unknown feldspar, and none in the other component of the mixture *i.e.*, andesine. (The mixtures analyzed contained not more than 1 per cent of foreign minerals such as biotite, corundum, etc.) From the BaO content found by analysis the percentage of BaO was calculated in a sample which would consist of 100 per cent of barium feldspar. The amount of celsian equivalent to the percentage of barium present was then computed. The theoretical barium content of celsian was used as a standard. The celsian content of the unknown barium feldspar, thus computed, proved to be 14.5 per cent, or very close to 15 per cent, a figure which corresponds to the composition indicated by its refractive indices, and indicates that it is an intermediate type between Winchell's hyalophane (Cn_{10}) and (Cn_{20}). In making the computations a deduction was made for the amount of hyalophane which might possibly occur in solid solution with the associated andesine. This correction is included in Table 4.

A standard specific gravity determination was made on the 2.686 g. fraction separated in bromoform. This was done by employing the pycnometer method; boiling the fragments in water, removing the air and making up to volume at 20°C. The value obtained was 2.697, which is slightly higher than that of the liquid in which the grains just floated (2.686), and indicates the inadvisability of depending on heavy solutions for accurate gravity determinations.

The barium feldspar in the central portion of the dike, (types *E* and *F*) was identified as a more alkaline type of hyalophane ($Or_{90}Cn_{10}$) than that found associated with andesine in the border phase. This was done by checking the refractive indices with those given by Winchell, and verifying by a test for barium on a representative sample without making a detailed separation as above. Optical and chemical tests were made on a fraction which floated in bromoform of sp. gr. 2.675. The refractive indices and specific gravity of the two types of hyalophane present in the dike rock are shown in the following table. Three types listed by Winchell have been inserted for the sake of comparison.

TABLE 5. PHYSICAL PROPERTIES OF HYALOPHANE

Composition	$Or_{90}Cn_{10}$		$Or_{85}Cn_{15}$		$Or_{80}Cn_{20}$
Type	A	B	A	C	A
Optic Sign	—	—		—	—
N_g	1.534	1.532	not	1.539	1.541
N_m	1.531	1.530	given	1.535	1.538
N_p	1.528	1.527		1.532	1.535
Sp. Gr.	2.65	2.675		2.697	2.73

A. Taken from Winchell, *loc. cit.*, 360.

B. Type found in central portion of dike (types *E* and *F*).

C. Type found in border portion of dike (types *C* and *D*).

Index determinations were made with the immersion method in white light. Because of poor cleavage traces the extinction angles of the hyalophane could not be determined with any degree of certainty. The mineral exhibited no signs of twinning when tested on the universal stage.

ACKNOWLEDGMENT

To those whose generosity and invaluable assistance made possible the completion of this study we extend our sincerest thanks. They are Dr. Edward H. Watson and Dr. Dorothy Wyckoff of the Department of Geology at Bryn Mawr College; Dr. Jermain H. Creighton and Dr. Walter B. Keighton of the Department of Chemistry and Miss Ethel Stilz, House Director, of Swarthmore College; and Mr. A. Williams Postel of the Department of Geology, University of Pennsylvania.

SELECTIVE STAINING TO FACILITATE ROSIWAL ANALYSIS

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Introduction

There is a recent tendency in the geological sciences to introduce the quantitative factor in laboratory investigation. In petrography the development of the micrometer stage and more recently the development of electric counters for thin-section analysis[1] has greatly facilitated the modal determination of rocks. Larsen and Miller[2] have studied the accuracy and limitations of Rosiwal analyses and outlined the conditions as to grain size, area of thin section and number of traverses, under which the mode of a rock can be determined by the Rosiwal method with an accuracy of 1 per cent.

If the constituent minerals can be rapidly and accurately recognized without repeated optical tests a Rosiwal analysis can be made with an electric counter in about 35 minutes. If, on the other hand, it is necessary to stop and identify individual grains, both the speed and the accuracy of the method are reduced.

Among the common rock-forming minerals, untwinned plagioclase and potassium feldspar are in some cases difficult to distinguish from one another and from quartz by inspection. In the undersaturated rocks nepheline may present similar difficulties of rapid identification and distinction from untwinned feldspar. A combination of staining methods is here presented which solves this difficulty and thus allows increased speed and accuracy of Rosiwal determinations.

Combined Staining Methods

The proposed staining technique is based on two previously described methods for staining nepheline and potassium feldspar, respectively. So far as is known the two stains have not previously been used in conjunction.

The nepheline stain involves gelatinization with hydrochloric acid, followed by coloring with an organic dye which is adsorbed by the silica gel. This method is described in several textbooks of petrographic methods, but the original reference is unknown.

The potassium feldspar stain depends upon decomposition of the sur-

[1] Hurlbut, C. S., An electric counter for thin-section analysis: *Am. Jour. Sc.*, **237**, 253 (1939).

[2] Larsen, E. S., and Miller, F. S., The Rosiwal method and the modal determination of rocks: *Am. Mineral.*, **20**, 260 (1935).

face with hydrofluoric acid, followed by staining with a solution of sodium cobaltinitrite, a potassium test solution. This method was described by Gabriel and Cox,[3] for use principally in distinguishing between potassium and plagioclase feldspar in granular material. Slight modification of their procedure was found necessary to adapt the method for thin sections. Tests were made on nepheline-bearing alkaline rocks, metamorphic schists, and on syenitic and granitic rocks from the Bancroft area in Ontario. It was found that these could all be successfully stained without change in the reagents or timing, but, as noted below, the procedure for quartz-bearing rocks omits the nepheline stain.

Procedure

(1) A preliminary microscopic examination of the thin section is made and the minerals identified qualitatively. It is advisable to make any necessary optical determinations before staining, since the treatment partially obscures the optical properties of some minerals.

(2) Stains are applied to thin sections which have been prepared without cover glasses, or to sections from which the covers have been removed. In the latter case it is necessary to thoroughly clean the surface of the rock section, first with xylol and then with alcohol.

(3) The exposed glass of the slide, (not the rock section itself), is covered with a thin coating of celluloid, (solution 1)[4] to protect the glass from hydrofluoric acid during subsequent fuming. The celluloid solution is applied with a small camel's-hair brush.

(4) For Nepheline-Bearing Rocks: The section is laid flat on a table and covered with concentrated hydrochloric acid, applied with a pipette. The acid is allowed to stand for *4 minutes* and is then washed off gently by repeatedly dipping the section in water. Care must be exercised to avoid washing away the gelatinous silica, which is not held tenaciously to the nepheline.

(5) While the section is still wet it is covered with an aqueous solution of malachite green, (solution 2), applied with a pipette. This is allowed to stand for *50 to 60 seconds* and is then washed off (again gently), by dipping in water. At this stage nepheline is stained a strong green.

(6) The section is now dried thoroughly for 24 hours. It should not be heated, as this tends to cause the stained silica gel to chip and come off. If the section is damp at the hydrofluoric acid-fuming stage it will be completely decomposed.

(7) The dried section is set in a lead tray and placed in the lead fuming

[3] Gabriel, A., and Cox, E. P., *Am. Mineral.*, **14**, 290 (1929).

[4] Reagents and apparatus are described at the end of the article.

box (apparatus 1), and exposed to hydrofluoric acid fumes at 65°C. for *50 seconds*. The green color of the nepheline stain is destroyed at this stage, but is brought back during subsequent treatment with the feldspar stain.

(8) After fuming, the section is covered with the feldspar stain (solution 3), applied with a pipette, and allowed to stand for *30 to 40 seconds*. Potassium feldspar is stained a strong yellow.

(9) Finally, the section is again washed carefully and dried slowly, and a cover glass is cemented on with liquid Canada balsam. Stained sections should not be heated when applying the cover glass as they are quite fragile and will break up if the lower balsam layer becomes soft. If the cover is applied with liquid balsam (not heated), the balsam remains sticky for a few days but the sections can be used almost immediately.

The above procedure leaves potassium feldspar yellow, nepheline greenish blue, and plagioclase feldspar remains uncolored. The staining treatment obscures the optical properties of nepheline, but optical determinations can still be made on the feldspars. The colors of both stains appear to be permanent on covered thin sections; no change was observed in stained sections after three months.

For Quartz-Bearing Rocks, or for rocks which contain neither quartz nor a feldspathoid, steps (4) and (5) above are omitted. The resultant potassium feldspar stain is the same. In addition, the surface of plagioclase feldspar is roughened and its apparent relief in thin section is raised so that untwinned plagioclase can be distinguished at a glance from quartz, which remains clear.

Application of the Method to Other Minerals

The same staining procedure, omitting steps (4) and (5), offers a method for distinguishing in thin section between leucite and analcite, minerals which are not easily identified by optical methods. Under the hydrofluoric acid and sodium cobaltinitrite treatment, with timing as for the feldspars, leucite is stained yellow and analcite remains uncolored.

This procedure also distinguishes alunite from kaolin, talc, sericite and brucite. This was not tested in thin section, but on a polished surface alunite is the only one of this group of minerals to show the yellow stain. It might be expected that sericite would also be stained, since the yellow precipitate depends upon the presence of potassium. This is not the case, however, since the micas, unless altered, are not sufficiently decomposed by the hydrofluoric acid treatment to give an appreciable stain with sodium cobaltinitrite. This behavior of the micas under the staining treatment was noted by Gabriel and Cox.

Other minerals tested in the rocks were:—hornblende, pyroxene, biotite, corundum, ilmenite, magnetite and calcite. The first five of these are inappreciably affected by the combined staining procedure, and their recognition is not impaired. Magnetite is almost completely dissolved and calcite completely dissolved by the HCl treatment, but enough magnetite remains so that the grains can be measured in Rosiwal analysis.

Further possible applications of these two stains may be suggested by the following notes—(minerals not specifically tested for the present investigation). In addition to the minerals referred to, the following dissolve in hydrochloric acid with surface gelatinization:—cancrinite, chondrodite, chrysotile, melilite, olivine and the zeolites.

The following dissolve in HCl without surface gelatinization: apatite, chlorite, cordierite (partly), epidote (partly), gypsum, sodalite, vesuvianite (partly), and the calcic members of the scapolite series.

The following may be expected to be inappreciably affected by the combined staining procedure: andalusite, axinite, beryl, goethite, garnet, hematite, kyanite, ottrelite, rutile, sillimanite, staurolite, sphene, tourmaline, zircon and zoisite. Hematite and goethite are slowly attacked by HCl.

Apparatus and Reagents

Apparatus 1—A much simpler fuming box than that described by Gabriel and Cox is suitable for thin sections, polished rock chips or granular material. This can easily be made by folding one-sixteenth inch lead sheet into a rectangular box two inches by three inches in plan dimensions, and three-quarters of an inch deep. A shallow tray and stand and a flat cover for the fume box can also be made from lead sheet without special equipment (Fig. 1). In use, concentrated hydrofluoric acid is poured in the box to a depth of about one-eighth of an inch and the box is heated from below with a low bunsen flame to about 65°C.

Solution 1 (protective coating). Sheet celluloid dissolved in acetone to about the consistency of glycerine.

Solution 2 (malachite green). Prepared by dissolving one gram of malachite green in 200 cc. of distilled water.

Solution 3 (potassium stain). Concentrated sodium cobaltinitrite solution, prepared by adding 15 cc. of glacial acetic acid and 25 cc. of water to 12.5 grams of $Co(NO_3)_2 \cdot 6H_2O$ and 20 grams of $NaNO_2$. Upon standing a yellow precipitate is developed in this solution. The solution is apparently stable in contact with the precipitate and should not be filtered.

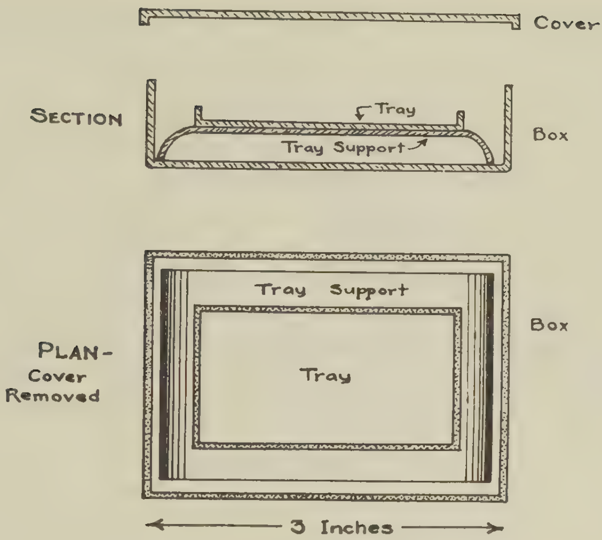

Fig. 1

Further Applications

The staining technique is considered to have its most important application in distinguishing minerals rapidly for Rosiwal analysis, but it may be found useful in some cases for mineral identification where optical methods are not entirely satisfactory, and also for facilitating study of the textural relations of rock minerals. For example, the potassium feldspar stain brings out perthitic intergrowths of the feldspars in a striking fashion. Both the gelatinization stain and the cobaltinitrite stain can be applied to polished rock chips as well as to thin sections. Rock chips should be heated, however, before being placed in the fuming box, otherwise the hydrofluoric acid will condense on the rock and the resultant yellow precipitate from the staining solution will be smeared over the surface.

It is difficult to illuminate stained polished surfaces well enough to permit these to take the place of thin sections for Rosiwal analysis. Mineral proportions can be estimated from stained polished sections, however, and the textural relations of a medium or coarse grained rock can be observed with a hand lens or binocular microscope.

FORMULA AND STRUCTURE OF RALSTONITE

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The rare fluoride, ralstonite, is found in small, clear, colorless octahedrons with cryolite and thomsenolite at Ivigtut, Greenland. It has also been reported from Colorado and Japan, but only the Greenland material has been analyzed. The best analyses of ralstonite are those of Brandl[1] and of Penfield and Harper.[2] From these analyses Penfield and Harper calculated the formula, $(Mg,Na_2)Al_3(F\text{-}OH)_{11}\cdot 2H_2O$.

Table 1 shows these two analyses together with recalculations considering part of the water to represent (OH) in order to satisfy valence requirements. The figures of the first column give Brandl's analysis as

Table 1. Analyses of Ralstonite

	1	2	1*a*	2*a*
Na	5.05	4.27	5.05	4.27
Mg	3.90	4.39	3.90	4.39
Al	23.06	24.25	23.06	24.25
F	57.68	39.91	57.68	39.91
OH			1.19	19.46
H_2O	10.17	18.73	9.56	8.43
	99.85	91.55	100.43	100.71

1. Analysis by Brandl, as corrected by Penfield and Harper.
2. Analysis by Penfield and Harper.

1*a* and 2*a*. Analyses 1 and 2 recalculated to account for the presence of OH.

corrected by Penfield and Harper for thomsenolite impurity. The figures in the last column differ slightly from those calculated by Penfield and Harper, partly because they apparently used different atomic weights and partly because their calculation does not exactly fulfill valence requirements.

Cell Contents

Thanks to the kindness of Mr. Dore of the Division of Plant Nutrition, University of California, the writer obtained powder, Laue and rotation x-ray diffraction photographs of ralstonite from Greenland. The material used was from the collections of the Department of Geological Sciences, University of California.

[1] Brandl, J., Ueber die chemische Zusammensetzung der Mineralien der Kryolithgruppe: *Liebig's Annalen der Chemie,* **213,** 1–15 (1882).

[2] Penfield, S. L., and Harper, D. N., On the chemical composition of ralstonite: *Am. Jour. Sci.,* (3) **32,** 380–386 (1886).

Table 2 gives the data for a powder photograph made with Mo radiation, using a zirconium filter and a cassette of 8 inch radius, calibrated

TABLE 2. POWDER PHOTOGRAPH OF RALSTONITE FROM GREENLAND. MO-RADIATION. CASSETTE RADIUS 8 INCHES

Estimated intensity	$\sin^2\theta$	$(h^2+k^2+l^2)\times q$	*hkl*
10	0.00388	3×0.001294	111
8	0.01422	11×0.001293	311
7	0.01554	12×0.001295	222
1			400
2	0.02452	19×0.001291	331
4	0.03106	24×0.001293	422
6	0.03549	27×0.001299	333, 511
7	0.04137	32×0.001292	440
1	0.04524	35×0.001293	531
1	0.05194	40×0.001298	620
4 (broad)	0.05682	43 44×0.001292	533 622
1	0.06206	48×0.001293	444
3	0.06591	51×0.001293	711, 551
3	0.07625	59×0.001293	731, 553
1	0.08306	64×0.001298	800
2	0.09340	72×0.001298	822, 660
1	0.09859	76×0.001298	662
1	0.10321	80×0.001292	840

by a comparison pattern with halite. This leads to a lattice constant of 9.87Å and to a cell volume of 961.5Å.[3] A cell of these dimensions does not contain an integral number of formular units, if the hitherto accepted formula is used. Instead Table 3 shows the census of the cell contents for both analyses assuming a density of 2.61. In the case of Pen-

TABLE 3. CONTENTS OF UNIT CELL OF RALSTONITE

	1		2			
	Sp. Gr. 2.61		Sp. Gr. 2.61		Sp. Gr. 2.56	
Na	3.25		2.82		2.76	
Mg	2.45	15.45	2.75	16.45	2.70	16.13
Al	13.00		13.70		13.43	
F	46.30	47.37	32.00	49.42	31.35	48.44
OH	1.07		17.42		17.09	
H_2O	8.08		7.14		7.00	

field and Harper's analysis the results are shown also for an assumed density of 2.56, the figure they reported for their best material. It is probable that the density of ralstonite is variable and that the material

of the second analysis, having more fluorine replaced by hydroxyl and having, as will be shown, some vacancies in its water positions, is actually of lower density. The table shows that the number of Al plus Mg in the cell is nearly 16, and the number of F plus OH is nearly 48. The sum of Na and Mg, on the other hand is between 5 and 6, and does not correspond to any number of equivalent points possible in a cubic cell.

Space Group

The Laue symmetry of ralstonite is O_h. Both powder and rotation patterns show only lines compatible with a face centered lattice. Space groups requiring the extinction of *hhl* when *h* is odd are excluded by the appearance of 113, 115 and other similar lines. This leaves five possible space groups, T_d^2, O^3, O^4, O_h^5 and O_h^7. The lines *hk*0 are found only with $h+k=4n$ (Table 6). Of the possible space groups only the last mentioned requires this restriction. It may be concluded that O_h^7-Fd3m is most probably the space group of ralstonite.

Structure

Proceeding from the figures of column 3 of Table 3, it is necessary to accommodate 48 (F, OH), 16 (Al, Mg), 2.8 Na and 7 H_2O in the unit cell. In the space group O_h^7 there is but one 48-fold position, $x00$. This will serve for (F, OH). There are two 16-fold positions, (*c*) $\frac{1}{8},\frac{1}{8},\frac{1}{8}$ and (*d*) $\frac{5}{8},\frac{5}{8},\frac{5}{8}$, but they are similar and the choice of one or the other for (Al, Mg) is equivalent to a choice of origin, so that 16 (*c*) may be taken arbitrarily for Al and Mg. A preliminary choice of the parameter for (F, OH) may now be made from the known radii of Al and F. If this is done it is found that neither Na nor H_2O can be fitted into the position 8: (*a*) 000. This leads to the following possible arrangement:—

48	(F, OH)	in 48: (*f*) $x, 0, 0$;
16	(Al, Mg)	in 16: (*c*) $\frac{1}{8}, \frac{1}{8}, \frac{1}{8}$;
2.8	Na	in 16: (*d*) $\frac{5}{8}, \frac{5}{8}, \frac{5}{8}$;
7	H_2O	in 8: (*b*) $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$;

both the last two positions being only partly occupied. This arrangement is the type $E8_1$ (*Strukturbericht*, **II**, 1928–1932), the structure type of pyrochlore and related minerals.

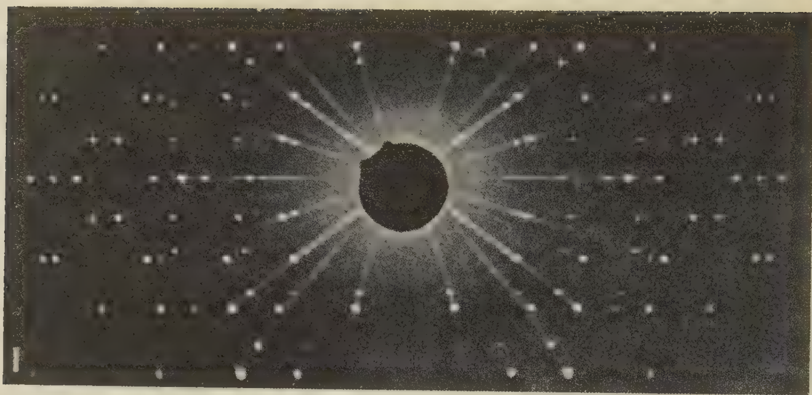
Table 4 shows the comparison of calculated and observed intensities of the first 22 possible lines in a powder pattern of ralstonite. From this it may be seen that the parameter of the (F, OH) is 0.19, the agreement of calculated and observed intensities being excellent for this value.

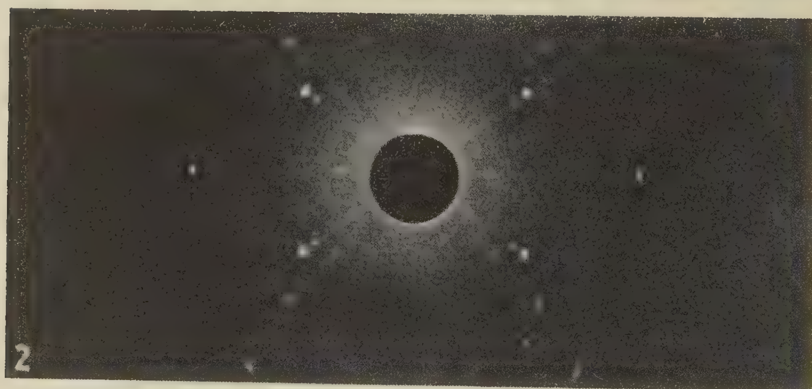
Table 4. Observed and Calculated Intensities of Lines in Powder Photograph of Ralstonite

	Observed		Calculated: Mg with Al in 8 (c)		Mg with Na in 8 (d)
hkl		*x* .20	.19	.18	.19
111	10	2.08	2.52	3.22	1.57
220	—	.07	.03	.01	.03
311	8	1.40	1.42	1.26	.80
222	7	1.10	1.10	1.10	1.10
400	1	.25	.15	.07	.15
331	2	.27	.32	.34	.13
422	4	.45	.53	.62	.53
511 }	6		1.11 { .04	.03	
333 }			1.07	1.11	
440	7		1.46	1.04	
531	1		.22	.16	
620	1		.27	.44	
533 }	4		.98 { .36	.48	
622 }			.62	.62	
444	1		.12	.19	
711 }	3		.62 { .06	.11	
551 }			.56	.38	
642	—		.02	.03	
731 }	3		.53 { .52	.33	
553 }			.01	.03	
800	1		.18	.19	
733	—		.06	.03	
822 }	2		.37 { .20	.14	
660 }			.17	.21	
751 }	—		.03 { .02	.00	
555 }			.01	.01	
662	1		.21	.20	
840	1		.24	.19	

Table 5. Interatomic Distances in Ralstonite

	Distances calculated from structure	Sum of radii
F–F (0, .19, 0)–(0, 0, .19)	2.65Å	2.66
F–F (.25, .06, .25)–(0, 0, .19)	2.60	2.66
F–(Al, Mg)	1.86	F–Al 1.90; F–Mg 2.11
F–Na	2.54	2.31
F–H_2O	3.06	2.65
Na–H_2O	2.14	2.30
F–(0, 0, 0)	1.88	
F–(x, x, x) $x=\frac{1}{8}$	1.86	





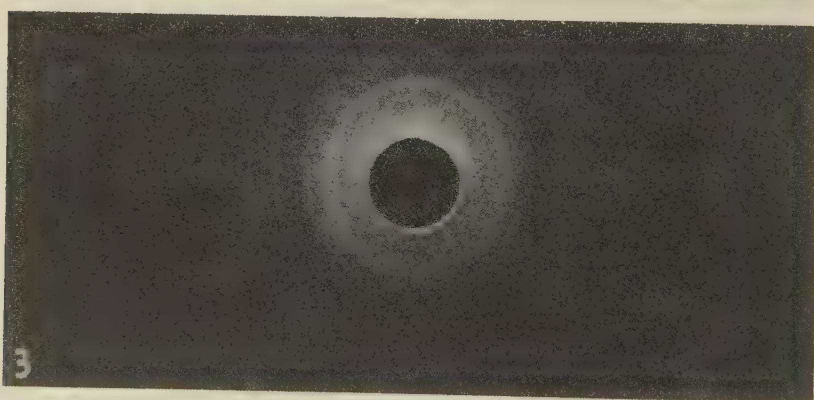


FIG. 1. Fresh ralstonite.

FIG. 2. Ralstonite heated to 365°. 27% loss.

FIG. 3. Ralstonite heated to 615°.

All rotation photographs on [001] with Cu radiation. Prepared in the Mineralogisches Institut, Tübingen.

The structure may be checked by reference to Table 5 which shows the interatomic distances compared with the distances corresponding to Goldschmidt's radii. Incidentally the distances to certain vacancies are also included to show that these are not suitable positions for H_2O or Na. The similarity of the two F–F distances shows that the coordination of F or of F and OH about Al or Mg is almost ideally octahedral. All values agree well with requirements except that Mg has rather a tight fit in the Al positions. The last column of Table 4 shows the calculated intensities considering that Mg and Na share a set of positions and that part of the set of positions occupied by Al is vacant. The intensity relations of the first few lines show unequivocally that Mg is not placed in the structure in this fashion.

Column 1 of Table 6 shows the summary of observations on a rotation pattern of ralstonite (Fig. 1). The indices and intensities of all spots are in agreement with the structure outlined above, except for the spot 442, which has intensity 2, whereas 424 is not observable. This spot should be absent due to extinctions required by the special positions occupied in this structure, though it is permitted by the space group. Other spots of this character, such as 644, 842 or 882 are not found on any of the films. A search on films of the related minerals, koppite, romeite and pyrrhite failed to reveal 442 or similar spots. A rotation pattern of ralstonite with Fe radiation also showed the spot 442, but not 424. In short, the presence of this disturbing spot was not to be denied in spite of its absence, as required, on the films of other materials of supposely analogous structure.

TABLE 6. INDEXING OF ROTATION PATTERNS OF FRESH (1) AND OF HEATED (2) RALSTONITE

*hk*0	1	2	*hk*2	1	2	*hk*4	1	2
200		5*	202		5	204		3*
220		4	222	10	10	224	7	2
400	4	3	402		4*	404	9	8
420		5*	422	8	3	424		2*
440	10	10	442	2†		444	6	3
600		3*	602	5				
620	5		622	9	5	624	1	
640		5*	642	1		644		1*
800	6	4				804	4	1
			822	7				
660	4	1	662	6	3	664	3	
840	5	3				844	5	1
			10·0·2, 862	2	1			
10·2·0	3	1	10·2·2	5				
880	3	1	882		1?*			
10·6·0	4		10·6·2	5				
			12·2·2					

*hk*1			*hk*3		
111	9	5	113	10	4
311	10	5	313	8	7
331	7	5	333	10	7
511	2	5	513	4	1
531	4	2	533	8	2
711, 551	6		713, 553	5	
731	6		733	3	1
751	1		753	2	
911	3		913	4	
931	4		933	1	
771	1?		773	4	
951	2		953	2	
			11·1·3	3	
11·3·1	4		11·3·3	5	
11·5·1	6				

* Spots not compatible with ralstonite structure, see text.
† Spot due to indirect diffraction, see text.

Several modifications of the structure which might yield an explanation of this spot were tried but failed to give satisfactory results. Finally it seemed that the spot must be due to indirect diffraction, "Umweganregung."[3]

[3] Renninger, M., "Umweganregung," eine bisher unbeachtete Wechselwirkungserscheinung bei Raumgitterinterferenzen: *Zeits. Physik*, **106**, 141–176 (1937) and Brandenberger, E., Umweganregungen und Auslöschungsgesetze: *Zeits. Krist*, **97**, 476–484 (1937).

It has been shown that the diffraction $h_1k_1l_1$ may be simulated under certain conditions if an x-ray beam which has been diffracted by $h_3k_3l_3$ suffers a second diffraction by $h_2k_2l_2$ within the crystal. In order that this should occur it is necessary that:—

1. $h_1=h_2+h_3$, $k_1=k_2+k_3$, $l_1=l_2+l_3$; where $h_2k_2l_2$ and $h_3k_3l_3$ are permitted lines for the structure;
2. $h_1k_1l_1$ and $h_3k_3l_3$ must reach the diffraction position with the incident beam simultaneously;
3. finally $h_2k_2l_2$ and $h_3k_3l_3$ must themselves yield strong diffractions and the crystal must be sufficiently large and perfect in order that a measurable intensity of $h_1k_1l_1$ shall result.

In the present case all of these conditions are fulfilled. It was first found graphically with the aid of a reciprocal lattice projection and then checked by calculation that both for Fe and for Cu radiation 333 reaches the diffraction position simultaneously with 442 upon rotation on [001]. The diffraction spot 442 on the films then arises from the secondary beam 333 which has suffered a further diffraction on $11\bar{1}$. It reaches a notable intensity because of the perfection of the crystal and because 333 and $11\bar{1}$ are among the strongest diffractions. This explanation of 442 is in harmony with the absence of 424. If 442 were a normal diffraction 424 should appear with about double its intensity. If 442 is merely an indirect diffraction via 333 and $11\bar{1}$, 424 should be absent as it does not reach the diffraction position simultaneously with 442 and 333, *i.e.*, condition 2 is not fulfilled.

Dehydration and Decomposition

In view of the variable water content an attempt was made to trace the effect of dehydration on the structure of ralstonite. Because of the small amount of material available and the difficulty of separating it from the accompanying minerals the number of dehydration experiments was somewhat limited. A 14 mg. portion of ralstonite showed little loss at 245°C. After brief heating at 445° a loss of 19% was recorded. Since this is about the maximum amount of water found in ralstonite, the heating experiment was broken off at this point. The crystals, though rather cloudy, had lost entirely the anomalous double refraction invariably found in fresh ralstonite. The index of refraction had dropped from 1.43 to about 1.39 and the density from 2.6 to 2.3. A rotation pattern showed that the structure remained intact and that the lattice constant was but little changed. This is most surprising, for the loss of 19% indicates that not only the water of crystallization was driven off but also hydroxyl water. In order that this should occur one might suppose that

certain of the 48 (f) positions are now vacated and others, formerly occupied by OH^{-1} are now occupied by O^{-2}.

Unfortunately the material used for this dehydration experiment was used up in tests for lattice constant, density and index of refraction. A new portion of 20 mg. was used for a repetition of the experiment. Again no notable loss was observed at 245°. This time the heating was carried on by more stages with the result that the crystals after 3 hours at 365° showed a loss of 27% and finally after further 4 hours at 450° reached constant weight with a total loss of about 30%. Initially clear, colorless crystals which have suffered this great loss are merely whitened and assume a porcelaneous appearance. The faces remain perfect reflecting surfaces as shown by repeated examination on the two-circle goniometer. Again a rotation photograph shows that the structure is largely maintained, as may be seen from a comparison of Figs. 1 and 2. The appearance of rings in connection with some of the spots and the "fuzziness" of the second photograph suggest the beginning of transformation to the unoriented aggregate which is obtained at still higher temperatures.

Table 6 shows the intensities and indices of spots observed on a rotation photograph of ralstonite which had been heated to 365° with a loss of weight of 27%, in parallel columns with like data for fresh ralstonite. The main features of the two patterns are similar but there are some notable changes in intensities and 8 or 9 new spots appear that are incompatible with the structure of ralstonite and with the space group O_h^7. Even though the dimensions are unchanged and the lattice remains face centered it may be concluded that there is extensive rearrangement of the remaining constituents when the volatiles are driven off from ralstonite.

Since the loss is more than the maximum amount of water known to occur in ralstonite it seemed certain that some other constituent must have been driven off by prolonged heating at 365 to 450°. To test this about 5 mg. of fresh ralstonite were sealed in a small evacuated glass tube and heated to 375° for about 2 hours. This resulted in the etching of the glass tube and the formation of a fine white powdery deposit, thought to be H_2SiF_6. This was confirmed by dissolving the white powder in water and obtaining a positive test for F ion.

One is now forced to the conclusion that HF may be driven off from ralstonite at moderate temperature without destruction of the crystal lattice. If, in the case of ralstonite having the composition shown in column 2*a* of Table 1 all the water is driven off and the H of OH goes off with F as HF, a total loss of 31.3% would result. Some such change has occurred in the ralstonite whose diffraction pattern is shown in Fig. 2.

Further heating of ralstonite for 3 hours at 615° causes no further loss

in weight. The material remains cloudy and isotropic and assumes a refractive index of about 1.57. The density varies slightly from fragment to fragment, but is near 2.4. The crystals remain outwardly perfect and the faces still yield such good reflections that the [001] axis may be accurately adjusted as rotation axis. Figure 3 shows the rotation photograph of such a "crystal." The smoothness of the rings corresponds to the appearance of a powder photograph, but slight intensification of a few of the lines where they cross the former position of layer lines suggests the last traces of common orientation. It appears at first glance that some of the lines may be indexed to correspond to the single crystal photographs, but this is not the case. Half of the lines do not correspond to any spots in the other patterns and the two strongest spots, 222 and 440 of Fig. 2 are not found in the powder pattern. It must be concluded that one or more new phases have been formed.

Relations to Similar Structures

In Table 7 are summarized the data on the occupation of the several lattice positions in minerals and compounds of the structure type $E8_1$. The parameter x has in some cases been changed for $\frac{1}{2}$-x and the positions 16 (c) and 16 (d) as well as 8 (a) and 8 (b) interchanged to agree with the choice of origin adopted for pyrochlore by von Gaertner and used in this paper for ralstonite.

No numerical values are given for the disposition of metals in the structures of minerals quoted from other authors because the determinations were in part on material that had been metamict or for which reliable detailed analyses were lacking.

Both Machatschki and Reuning observe that the materials they examined show a deficiency of positive ions for full occupation of the lattice points so that vacancies may occur, say in the 16 (c) positions, but the variable valence of some of the metals in this group introduces a measure of uncertainty. In Sb_3O_6OH, $BiTaO_6F$ and in ralstonite partly vacant sets of positions are definitely established.

The formula $X_2Y_2(O, OH, F)_7$ has been used for minerals with pyrochlore structure, there being eight such units in the cell. Rosen and Westgren[4] object to this formula on the ground that $Ca_2Ta_2O_7$ is not isomorphous with this group. They prefer the formula $X_2Y_2O_6(OH, F)$ with no oxygen in the eight-fold position. von Gaertner originally placed (O, F) in 48 (f) and (F, O) in 8 (b). Unfortunately no consideration of intensities can establish the distribution of O, OH, F and H_2O over these two positions.

[4] Rosen, O., and Westgren, A., Minerals of the Varuträsk pegmatite XII. On the structure and composition of minerals belonging to the pyrochlore-atopite group and an x-ray analysis of disintegrated stibio-microlite: *Geol. Fören. Förhandl.*, **60**, 216–235 (1938).

TABLE 7. MINERALS AND ARTIFICIAL COMPOUNDS OF THE PYROCHLORE GROUP

Name or formula	Lattice constant	16 (*c*)	16 (*d*)	48 (*f*) $x00$	x	8 (*b*) $\frac{1}{8}\frac{1}{8}\frac{1}{8}$	Reference
Pyrochlore synthetic	10.38Å	Nb	Na, Ca	O	.20	F	v. Gaertner
Pyrochlore natural	10.34	Nb, Ti, Ta	Ca, Na, Ce, etc.	O, F	.20	F, O	v. Gaertner
Koppite	10.37	Nb, Fe	Ca, Ce, Na, K	O	.19	O, OH, F	Brandenberger
Romeite*	10.26	Sb	Ca, Na, Mn	O	.20	OH(F)	Zedlitz
Schneebergite	10.30	Sb	Ca, Na, Fe	O		OH	Zedlitz
Lewisite	10.27	Sb, Ti	Ca, Fe, Na, Mn**	O, OH		***	Machatschki
Pyrrhite	10.41	Nb, Ta, Ti	Ca, Na, Fe	O		OH, F	Machatschki
Microlite	10.38	Ta, Nb	Ca, Na, Fe	O		OH, F	Reuning
Bindheimite	10.41	Sb	Pb	O		O	Hägele
Ralstonite	9.87	13Al, 3Mg	3Na	31F, 17OH	.19	$7H_2O$	
Sb_3O_6OH†	10.28	$16Sb^{V}$	$8Sb^{III}$	48 O	.21	8 OH	Dihlström and Westgren
$BiTa_2O_6F$	10.46	16Ta	8Bi	48 O	.21	8F	Dihlström and Westgren
$NaSbO_3$††	10.20	16Sb	16Na	48 O	.21		Schrewelius
$AgSbO_3$	10.23	16Sb	16Ag	48 O	.21		Schrewelius

* Atopite and weslienite are varieties of romeite.

** There are many vacancies in the 16 (*d*) position of lewisite.

*** The 8 (*b*) position of lewisite is practically unoccupied.

† Dihlström and Westgren consider that Sb_2O_4 or $Sb_2^{III}Sb_2^{V}O_8$ described by G. Natta and M. Baccaredda, Tetrossido di antimonio e atimoniati; *Zeits. Krist.*, **85**, 271–296 (1933), is really Sb_3O_6OH.

†† There also exists a rhombohedral form of this compound, isomorphous with ilmenite.

VON GAERTNER, H. R., Die Kristallstrukturen von Loparit und Pyrochlor: *Neues Jahrb. Min.*, **61A**, 1–30 (1930).

BRANDENBERGER, E., Die Kristallstruktur von Koppit: *Zeits. Krist.*, **76**, 322–334 (1931).

ZEDLITZ, O., Die Kristallstrukturen von Romeit und Schneebergit: *Zeits. Krist.*, **81**, 253–263 (1932).

MACHATSCHKI, F., Die Pyrochlor-Romeit-Gruppe: *Chemie der Erde*, **7**, 56–76 (1932).

REUNING, E., Mikrolithvarietäten von Donkerhuk, Südwestafrika: *Chemie der Erde*, **8**, 186–217 (1933).

HÄGELE, G., Röntgenographische Untersuchung des Bindheimits von Waitschach bei Hüttenberg, Kärnten: *Zentralbl. Min.*, **1937A**, 45–50.

DIHLSTRÖM K., and WESTERN, A., Über den Bau des sogenannten Antimontetroxyds und der damit isomorphen Verbindung $BiTa_2O_6F$: *Zeits. anorg. u. allgem. Chem.*, **235**, 153–160 (1937).

SCHREWELIUS, N., Röntgenuntersuchung der Verbindungen $NaSb(OH)_6$, $NaSbF_6$, $NaSbO_3$ und gleichartiger Stoffe: *Zeits. anorg. u. allgem. Chem.*, **238**, 241–254 (1938).

In many of the minerals of this group the intensity of the lines is largely controlled by the heavy Sb and Nb and related ions. With ralstonite no such difficulty arises and it is possible to show that certain positions are partly vacant. In lewisite the eight-fold position is likewise largely vacant and in $AgSbO_3$ and $NaSbO_3$ it is wholly unoccupied, suggesting that the pyrochlore structure may be stable in spite of wide variation in the number and character of negative ions and water molecules. The essential feature, common to all the examples listed in Table 7, is the coordination of negative ions in $x00$ about the position $\frac{1}{8},\frac{1}{8},\frac{1}{8}$, giving a continuous network of linked octahedrons.

The writer is very grateful to Professor F. Machatschki, director of the Mineralogisches Institut in Tübingen, where the work was concluded, for much encouragement and many helpful suggestions.

NEW METHOD FOR THE DETERMINATION OF FELDSPAR TWINS

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Abstract

Subject: Feldspar twins. (1) An appeal for their study from the point of view of their possible exploitation. Can they be used as a basis of igneous rock correlation? What decides which twin laws shall prevail? (2) A description is given of a new, very rapid procedure for the determination of the twin laws. We estimate the time needed for the determination of such twin laws by the new "five-axis method" to be definitely less than 10% of the time required for the original four-axis technique of Fedorov. There is also afforded an increase in both accuracy and range of applicability.

Our purpose is to describe a new method for the determination of feldspar twins—a method in which we have found combined (a) the speed of the Rittmann method without its attendant limitations mentioned below, (b) the accuracy of the Fedorov method at its best. The recognized points of weakness of the Fedorov method, mentioned below, have been found to offer no difficulty here. The great bulk of the Fedorov graphical procedures is eliminated. In fact we feel that this technique of feldspar-twin determination makes this type of study sufficiently rapid and simple to be thoroughly satisfactory for routine use.

The general situation of feldspar-twin study in America may profitably be reviewed as a background. There has been little published study of feldspar twinning in America. The reasons seem to be two—first, at best the method has been a little tedious and slightly involved; second, there has been no recognized practical application of the results to be obtained. The procedure presented in this paper, we believe, completely eliminates all reasonable involvement and tedium. The second question still remains and adequately justified limiting the general practice of the technique. However, a diligent search is now being conducted at Wisconsin hoping to answer several most obvious questions one may ask about the occurrence of feldspar twins. According to the results of these studies we shall either expand such work further or discontinue our part in it almost entirely.

An early concept prevailed that certain twin laws were to be found more prominently in calcic plagioclase and others largely in the more sodic plagioclase. On this basis, composition would be a dominant controlling factor in deciding the twin law. A preliminary study made in the senior writer's laboratory by Chapman (1) on a differentiated sill showed by statistical count that the same twin laws prevailed from bottom to top of the sill. If composition were a controlling factor, then

such uniformity would not be possible. Although one such study is not conclusive evidence, it helped to disprove an unfounded idea. It did not reveal the real basis of control of feldspar twinning, which still remains a mystery.

Without understanding this basis it is entirely possible that feldspar twinning may be of practical value, if used empirically. If one intrusive or one phase of one intrusive body is characterized by one twin pattern, then such a pattern may serve as a thumb print for that igneous rock—of value in correlation. Study is now in progress to attempt to answer this question.

We do not know the effect on feldspar twinning of metamorphism which involves the crystallization of feldspars. Such a study is also in progress.

Adjacent twin lamellae quite normally show a difference in anorthite content as revealed by optical study. We do not know if this is apparent or real, that is, due to strain or an actual chemical difference. We hope to answer this question shortly.

Other questions will occur to the reader according to his experience. These are mentioned in the hope that we may enlist the interest and cooperative effort of others in this most fascinating study. The consistent occurrence of feldspars in igneous rocks lends unusual importance to any discovery of twin significance. Our admitted ignorance in this field is a strong incentive. With a new, thoroughly practical method at hand to facilitate the work we hope that more American petrographers will join in this type of study.

The method suggested here is brief. We shall state it first and then illustrate it. Assume a single feldspar twin of two lamellae. Orient either one on the universal stage (2) (10). No graphical construction is needed on the five-axis stage and the procedure involves liberally five minutes in an average case. Record the oriented position. Now make the composition face vertical and north-south (as required for the Rittmann procedure) and rotate on the outer east-west axis till the lamellae are equally bright. Measure the extinction angle of each lamella. This reveals the position of the twinning axis. Record the new readings of the stage. A short graphical procedure, made on a stereogram, follows, namely—rotate the twinning axis from its oriented position just found to the position it occupied when the first lamella was oriented. This is most easily done by merely following the measured curves on a printed stereogram as illustrated below. The stereogram so constructed is now placed on plots already published in several places (3), and the twin law and feldspar composition are read off. Two illustrations follow.

Procedure for illustration 1. A rough sketch was made of the crystal and the extinction relationships of the various lamellae were noted. On the basis of these relationships the lamellae were grouped into three units (Fig. 1). First, unit 1 was oriented on the five-axis stage and the attitudes of the optic elements were determined. This was recorded in Table 1 (columns 1 and 2). Second, the composition face was made north-south and vertical by rotations on the outer vertical and north-south axes. Verticality is recognized when the line defining the composition face is finest. The new readings were recorded in the third column under composition face. Therefore the entry N-S 3°W means that the north-south axis has been rotated from 28°E to 3°W, a total of 31° west (to be used later). It is well to let all entries be direct readings from the universal stage in order to avoid confusion. The inner vertical axis should not be involved in this orientation of the composition face. The inner east-west axis should also be excluded whenever possible. Third, a wide rotation was made on the outer east-west axis, which is, of course, normal to the composition face. Two observations were made on this rotation. One, the relative intensities of illumination of units 1 and 3 were found to be constant. This twin relationship was thereby recognized as normal,* the axis being east-west and horizontal, that is, coinciding with the pole of the composition face. This is implied on the data sheet (Table 1) by the entry "normal." Two, the relative intensities of illumination of units 1 and 2 were found to vary. This twin relationship was thereby recognized as parallel or complex, the axis lying somewhere within the composition face. Two further steps locate this twinning axis, namely, one—the rotation on the outer east-west axis was repeated carefully to the position in which units 1 and 2 were equally bright (Fig. 2). The outer east-west axis was read and recorded as T_{1-2}, 24°S. The twinning axis was then known to be either vertical, or horizontal and north-south. Two—the crystal was rotated on the microscope stage to bring one or both of these units to extinction. The extinction values were found to be equal and opposite in sign, indicating the axis to be horizontal (8). This is recorded in the last column by the entry *H*. (If both units had gone to extinction simultaneously the final entry would be *V* instead of *H*, indicating that the axis was vertical.) All recordings for this crystal are now complete.

* Four convenient references are given where twin laws may be reviewed—(4), (5), (6), (7).

TABLE 1. TWIN DETERMINATION—FIVE-AXIS METHOD
Reference Data: G38–5A—Beaver Bay—L. Superior
Hemisphere 1.559. Zero Values: Mic. 359.8° O. V. 90°.

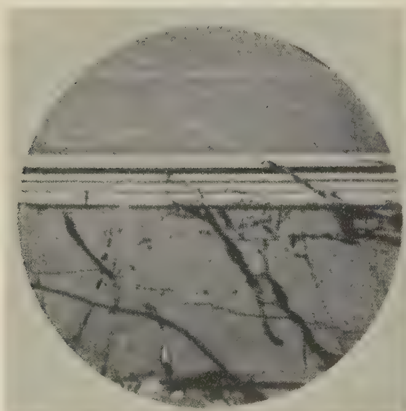
Unit 1:

I. E-W	8°S
N-S	28°E

I. V.	359.2
α	N
β	$\perp$
γ	E

Comp. Face 1.	O. V.	↶ 29°	
	N-S	3°W	
	I. E-W	—	
Twin Axes (O. E-W)	T_{1-3}	Normal	
	T_{1-2}	24°S	H

REMARKS: Composition Face (010)
Twin Laws. T_{1-2} Carlsbad
T_{1-3} Albite
Composition 79–81% An.



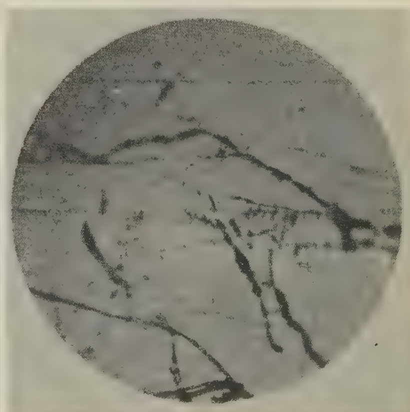
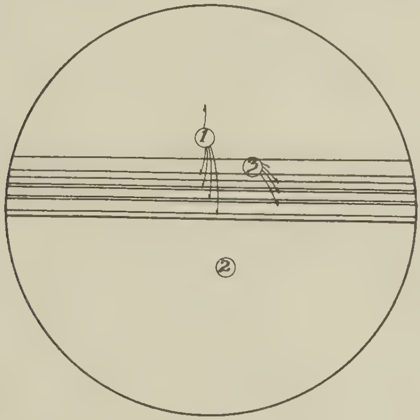


FIG. 1. The plagioclase crystal of example 1 showing the twin lamellae in contrasting illumination.

FIG. 2. The same crystal as that shown in Fig. 1 rotated to a position of equal illumination.

The accuracy of the position of the twinning axis, that is, the position of equal illumination obtained on the outer east-west axis, may be checked by the values of extinction on the microscope stage. These extinction values should be equal and either of the same or opposite sign. If they are not quite equal, then modify slightly the rotation on the outer east-west axis.

The accuracy of this position of the twinning axis may be enhanced also by another slight modification if the first rotation on the outer east-



Key to Figs. 1 and 2

west axis yielded a difficult observation. The position of equal illumination may be determined either when the composition face is north-south or in the 45° position. If the north-south position seems unfavorable, then the 45° position is to be preferred. Secure the 45° position by a rotation on the microscope stage—it is to be measured not estimated.

The remaining steps of the procedure are accomplished briefly on the stereographic projection. The purpose is to rotate the pole of the composition face, P, and the twinning axes T_{1-2} and T_{1-3} from the positions of known orientation back to the positions they occupied when unit 1 was oriented. This is done, of course, on the stereographic projection by reversing the rotations which led to the recordings of column three of the data sheet.*

* If hemispheres of index about 1.56 are used, corrections for the difference in index between crystal and hemisphere are negligible. If, however, other standard hemispheres are used, it is necessary to make these corrections for rotations on horizontal axes. The method of making these corrections has been discussed elsewhere (3), (9), (10).

The details for carrying out these rotations on a stereographic projection follow. (See Fig. 3.)* First—to locate the pole of the composition face, P, which is also the twinning axis, T_{1-3}. (a) Since the oriented pole was horizontal and east-west, the rotation of 29° counter clockwise was made as shown in Fig. 3; this brings the north-south axis into its true

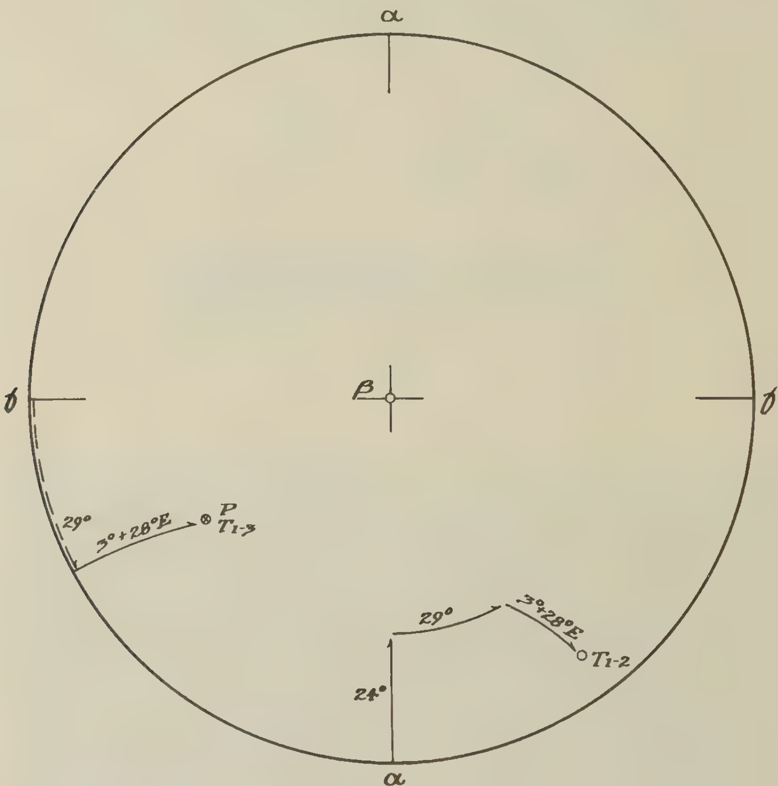


FIG. 3. The graphical rotations given in Table 1 employed to locate the pole and twinning axes on the plot of unit 1 in oriented position.

north-south position. (b) Since the north-south axis was rotated from the position 28°E to the position 3°W, a reversal requires a rotation of 31°E. This gives the position of the pole of the composition face and the normal twinning axis, T_{1-3} when unit 1 was oriented. Second—to locate

* The rotations are perhaps most simply made on a blank stereogram on which the cardinal orientation of unit 1 is indicated by placing α, β, and γ in their proper positions. This is superposed on some combination of Wolff and Fedorov nets.

the twinning axis T_{1-2}. (a) Since this axis was horizontal and north-south when oriented, and since the outer vertical axis was inclined by the rotation of 24°S on the outer east-west axis, therefore, the rotation of 24°N was made as shown in Fig. 3. This brings the outer vertical axis to its true vertical position. The remaining two rotations, 29° counter clockwise and 31° east were made as for the pole of the composition face. This is the position of T_{1-2} when unit 1 was oriented.

The projection thus completed may now be superposed on the proper Fedorov stereogram (3). The points P, T_{1-2} and T_{1-3} should fall reasonably close to the migration curves which in turn indicate the composition face, the twin laws, and the anorthite content. This crystal was found to have a composition face (010) and an anorthite content of 79–81%. T_{1-2} falls on the migration curve of the Carlsbad law and T_{1-3} the albite law. The graphical execution of this last step is reserved for the second example in Fig. 6.

Procedure for illustration 2. This second example is expanded to show that any number of twin relationships, true or theoretical, will be revealed by the positions of the twin axes on the Fedorov migration curves. The data for this crystal are recorded in Table 2. This crystal is illustrated in Fig. 4. Notice that only T_{1-3} and T_{3-4} for (010) and T_{1-2} in (001) have true twin relationships. The following twin relationships are regarded as purely theoretical since the lamellae represented have no mutual composition face: T_{2-3} and T_{1-4} in (010). T_{2-3} in (001) and the normal twin T_{2-4} for (001). In other words, it is to be borne in mind that any value that may accrue to this type of study lies wholly revealed by actual composition face relationships. Since the remaining theoretical relationships can be deduced inferentially, their measurement on the stage leads only to redundancy.

The details follow: As described for the first illustration, unit 1 (Fig. 4) chosen arbitrarily, was oriented and the details recorded in Table 2. Similarly, too, the most convenient composition face was brought to the required north-south and vertical orientation, and the new readings recorded as before. The inner vertical and the inner east-west axes remained unchanged. And again as before, a rotation was made on the outer east-west axis to distinguish the normally twinned lamellae; and a repeated rotation on this axis located the parallel and complex twinning axes. In completing this step by comparing the extinction angles of the respective units on the microscope stage, two of the twinning axes were found to be vertical—that is, the units went to extinction simultaneously. The other two recorded twinning axes introduced nothing which has not previously been discussed.

TABLE 2. TWIN DETERMINATION—FIVE-AXIS METHOD
Reference Data: WRC-37—Bridgland Tp., Ont.
Hemisphere 1.559. Zero Values: Mic. 0° O. V. 90°.

Unit 1:

I. E-W	37° S
N-S	35° W

I. V.	346
α	N
β	$\perp$
γ	E

Comp. Face 1.	O. V.	22.5°	
	N-S	55° W	
	I. E-W	—	
Twin Axes (O. E-W)	T_{1-3}	30°N	H
	T_{3-4}	Normal	
	T_{2-3}*	27°N	N
	T_{1-4}*	24°N	N

Comp. Face 2.	O. V.	77.5°	
	N-S	0°	
	I. E-W	19°N	
Twin Axes (O. E-W)	T_{1-2}	22°S	H
	T_{2-3}*	23°S	N
	T_{2-4}*	Normal	

REMARKS: Composition Planes 1) (010) 2) (001)
Composition 70% An.
Twin Laws: T_{1-3} in (010)-Carlsbad
$T_{3-4} \perp$ (010)-Albite
T_{1-2} in (001)-Acline

* For purposes of illustration the following theoretical twin relationships were determined on the stage. No actual composition face is represented. Instead (010) and (001) of the units were used. T_{2-3} and T_{1-4}-Albite-Carlsbad, in (010). T_{2-3} in (001)-Ala. T_{2-4} in (001) Manebach.

The other composition face (001) was next oriented, studied, and recorded with the following differences in the procedure. It was found inconvenient to make this composition face north-south and vertical by means of the north-south axis alone. The north-south axis was therefore

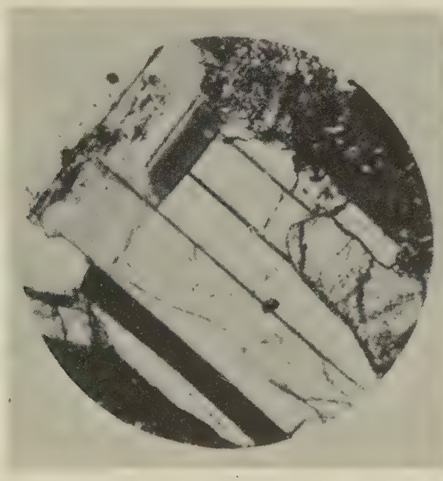
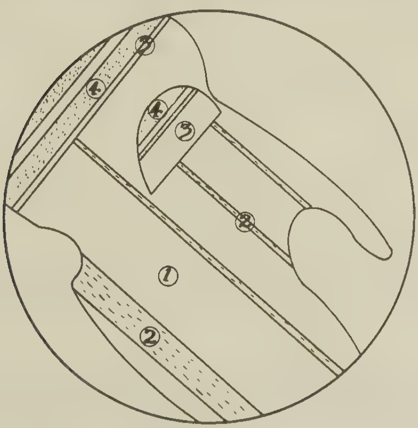


FIG. 4. The plagioclase crystal of example 2.



Key to Fig. 4

rotated to its zero position and the orientation was accomplished on the inner east-west axis in conjunction, of course, with the outer vertical axis. The north-south axis was reduced to its zero position to facilitate subsequent graphical rotations. When it is necessary to employ the inner east-west axis for this step, it is desirable, always, to set the north-south axis as done here.

Three twinning axes were located and recorded as before.

The graphical procedure again consists, as in illustration 1, of those steps which are necessary to bring the poles and the several twinning axes, each, from the position which it occupied when its orientation became known and recorded, back to its position when unit 1 was oriented. This was done as before by reversing the rotations which led to the entries in column 3, Table 2. We shall note mainly the differences introduced by the use of the inner east-west axis in orienting the composition face.

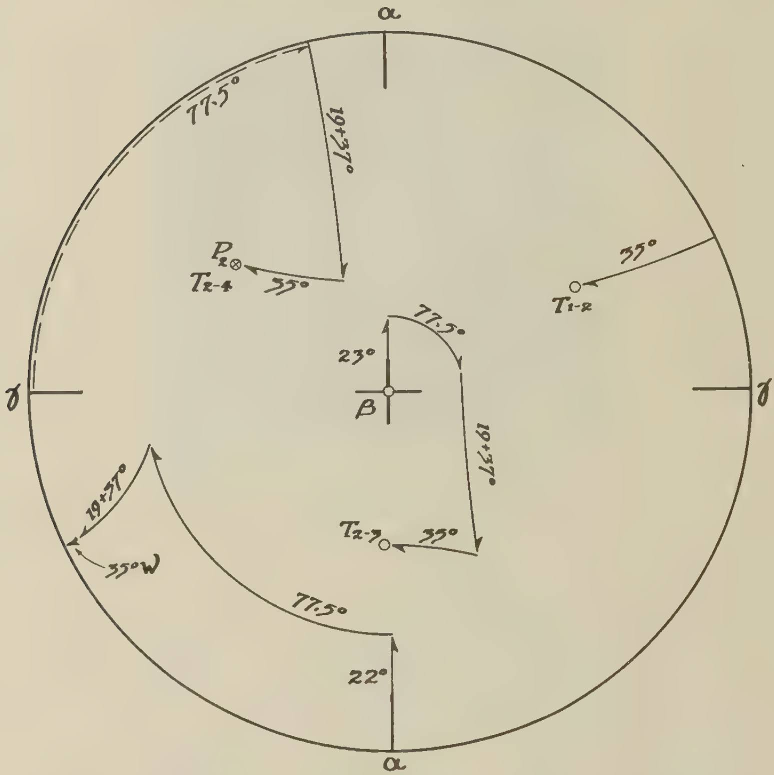


Fig. 5. The graphical rotations for the crystal of example 2 from the data of Table 2 employed to locate the pole and twinning axes for (001) on the plot of unit 1 in oriented position.

The order in which the graphical rotations are made alone needs explanation.* Consider T_{1-2}. From the oriented position of the twinning axis rotate first 22°N to make the inclined outer vertical axis vertical. See Fig. 5. Then rotate 77.5° clockwise (opposite to the direction recorded in Table 2) on the outer vertical axis to make the north-south axis north-south. Since the north-south axis was set at zero we may make next the rotation 19+37°S on the inner east-west axis. (If the north-south axis were at any other setting, it would be necessary to bring this value to zero before any graphical rotation could be made on the inner east-west axis.) Lastly, rotate west 35°,—the original setting for the north-south axis.

If an axis is vertical, the only difference in the procedure is that the rotations commence at the center of the stereogram.

The three rotations T_{1-2}, T_{2-3}, T_{2-4} are illustrated in Fig. 5.

The completed stereogram was superposed in proper orientation on the Fedorov migration curves as described in illustration 1 and the twin relationships read off. Figure 6 illustrates these two stereograms in proper superposition.† The twin laws are found to be T_{1-2}—acline, T_{2-3}—Ala, T_{2-4}—Manebach.

The procedure may be summarized.

1. Choose unit 1 arbitrarily and orient it. If there is any range of selection, then chose unit 1 for its quality of extinction (avoiding wavy or patchy extinction) and for its many contacts with other twin lamellae.
2. Make the most convenient composition face north-south and vertical.
3. Rotate on the outer east-west axis to sort out the normal and parallel or complex laws.
4. Rotate again on the outer east-west axis to equal illumination for the parallel or complex twins.
5. Determine the positions of the twin axes from the extinction angles.
6. Repeat steps 2–5 for other composition faces.
7. Rotate the recognized poles and twinning axes to the positions they occupied when unit 1 was oriented.
8. Superpose the completed drawing on the proper Fedorov stereogram to obtain the results.

* Students sometimes experience difficulty in determining the order of the graphical rotations, and an error here leads, of course, to complete failure. Since the Fedorov net provides for graphical rotations on only three mutually perpendicular axes, namely, north-south, east-west, and vertical, then each graphical rotation is possible only if the axis involved is graphically in one of these three cardinal positions. To illustrate—if a rotation is to be made on the inner east-west axis and the data indicate that the north-south, outer vertical, and outer east-west axes are inclined, then, since the inner east-west axis is dependent on (supported by) each of these, each of them must be reduced to its zero position before the inner east-west rotation may be made. Otherwise the inner east-west axis is inclined and the Fedorov net is not adapted. Furthermore, in bringing these supporting axes to zero, the same rules of order must be applied.

† The four possible positions of superposition are described elsewhere (3), (4), (5), (7).

To recapitulate: The advantages of this technique are: (1) It greatly shortens the time required for a determination. (2) Unlike the Rittmann method it may be applied to all twin laws. (3) Unlike the Rittmann method it is applicable to plagioclase of any composition, not merely the

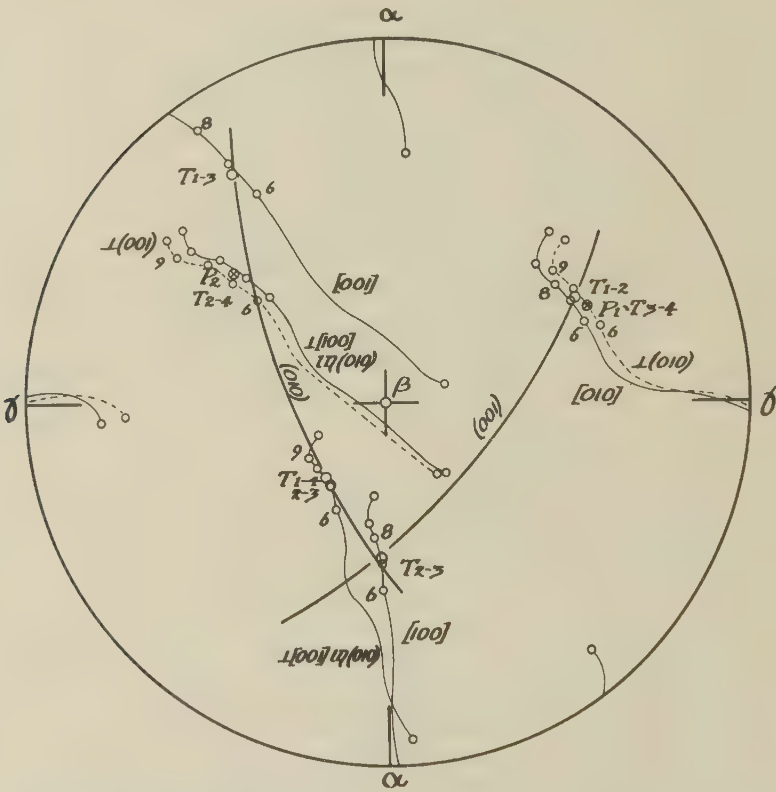


Fig. 6. Graphical representation of the crystal shown in Fig. 4 (in heavy lines) in superposition on the Fedorov migration curves of both the parallel and complex twinning axes (in light lines) and the poles of the composition faces (in broken lines). The migration curves are in inverted position instead of as customarily printed. This avoids the confusion of showing the plot inverted.

sodic two-thirds. (4) It will, like the Fedorov method, reveal a new twin law if present. (5) Unlike the Fedorov method it may be applied to those fine twin lamellae which are too narrow to be oriented optically. This method requires only that one lamella of one unit of a crystal be large enough to orient, and that the others be suitably visible under the microscope. (6) In that this method is based on the use of the Fedorov

migration curves, it sacrifices none of the completeness and accuracy of the Fedorov method. In that it eliminates much of the orientation procedure and most of the graphical procedures of the Fedorov method, it eliminates the greatest hazard of inaccuracy of that technique.

This method is applicable only on the five-axis universal stage. We refer to this, therefore, as the *Five Axis Method of Feldspar-Twin Determination.*

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ALTERATION OF GLASSES TO MONTMORILLONITE

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The formation of bentonite as the product of the geologic alteration of volcanic glass or ash has been postulated by many writers (4, 12, 14, 16, 18). Their conclusions have been based on the following facts: Thin sections of bentonite show the same structure as particles of glassy material often associated with it; the presence of feldspar and the absence of appreciable amounts of quartz; the presence of nearby alkaline salt deposits, and the chemical composition of the bentonite clay itself. Conditions for the formation of this clay and the range of its stability, however, have been investigated but cursorily, and it, therefore, should be of considerable geologic interest to determine the conditions under which bentonite may be obtained from volcanic glass or ash. This paper presents the results of an investigation of the alteration of glass to bentonite under the influence of high temperatures and pressures.

Most geologists consider the alteration of the glass or ash to have taken place after deposition, due to the leaching action of natural waters. On the other hand, it is probable that in the case of ash, the alteration may have taken place partially in the air, under the influence of volcanic and atmospheric gases. This can readily be imagined when one realizes that volcanic ash may be suspended in the air for considerable periods of time, as demonstrated by the famed eruption of Krakatau, where the ash circled the globe. The leaching process, however, is probably a much more important effect, in view of the fact that it has been proceeding since the deposition of the glass or ash.

Mineralogically, the essential mineral in bentonite is usually montmorillonite, less often beidellite (14). These are hydrous alumino-silicates having a chemical formula of $Al_2O_3 \cdot 4SiO_2 \cdot nH_2O$ and $Al_2O_3 \cdot 3SiO_2 \cdot mH_2O$, respectively, where the alumina in the latter is partially replaced by iron oxide. The former has been shown (1, 5, 6, 7) to consist of a three-layer lattice of silica, alumina and silica planes joined together by oxygen bridges, the unit crystals being held together by electrostatic forces (1). Swelling occurs when water is adsorbed between the unit layer parcels, thus forcing them apart. The swelling of montmorillonite occurs only with particles of colloidal size; Fuller's earth, which has been shown to contain a high percentage of montmorillonite (8), is generally non-colloidal in dispersions of its finest particles and non-swelling, although its adsorptive capacity is very high. Base-exchangeable ions are adsorbed either on the unsaturated oxygen bonds due to the fracture of

the unit crystals or to the unsaturated bonds created when an aluminum atom takes the place of a silicon in the silica layer. The aluminum layer may be partially or wholly replaced by iron or magnesium and occasionally by other elements (3, 10). Bentonites in the natural state contain varying amounts of colloidal material, depending on the state of decomposition of the glass, and the purity of the initial deposit of glass or ash.

Previous work on the synthesis of montmorillonite has been restricted entirely to the use of alumina and silica gels. Ewell and Insley (2) prepared beidellite from co-precipitated alumina and silica gels by heating them in an autoclave at 355°C. and 385°C., and found that the presence of soda was necessary for the reaction to take place. Somewhat later, Noll (13) prepared montmorillonite from silica and alumina gels by heating at 300°C. and 87 atmospheres, and found that the reaction took place in alkaline solutions of moderate concentrations. In acid or slightly alkaline solutions, kaolinite was formed. The latitude of concentrations was found to be much greater with $Mg(OH)_2$ than with the other alkalis. The use of gels, however, does not simulate the initial formation of montmorillonite from glass in nature, although some writers (15) have predicted the formation of an intermediate alumina and silica gel structure in the genesis of clay minerals.

Procedure

The reactions were carried out in a platinum crucible placed inside a reactor of the cap and case type, described by Morey and Ingerson (11). The powdered glass was placed in the crucible, the reagent added and the top placed on the crucible tightly, and the crucible placed in the reactor. 75 cc. of water were then added to give the desired pressure; the total volume of the reactor was 230 cc. Heating was accomplished in a thermostatically controlled furnace regulated to within ± 10°C. for times varying from 26 to 350 hours. The temperature selected for the reaction was 300°C.

The effect of reagent, time of heating, particle size of the glass and type of glass was investigated. The reagents used were H_2O, H_2SO_4, NaOH, KOH, $Ca(OH)_2$ and $Mg(OH)_2$. The glass was ground to pass 200 mesh in most of the runs; some of the glass was ground to less than 10 microns to establish the effect of particle size. A synthetic glass with a composition comparable to that of a Wyoming bentonite was made, and a natural obsidian or volcanic glass from Yellowstone National Park was obtained for use in these experiments.

The products obtained from the reactions were dispersed in water, allowed to sediment, and the colloidal fraction electrodialyzed to remove

extraneous salts which might confuse the x-ray patterns. Identification was made by means of x-ray diffraction patterns and dehydration curves.* The colloidal size of the clay particles made identification of the products by microscopic means impossible. In cases where montmorillonite was formed, dye adsorption tests were made with methylene blue whenever the quantity of material was sufficiently large. Comparison tests were made both on a Wyoming bentonite and on a California bentonite in which all the alumina is replaced by magnesia.

It should be noted here that these reactions differ in several respects from reactions carried out with gels such as reported by Noll, Ewell and Insley and others. With gels, we are dealing with matter present in a more or less degree of hydration, and a much greater mobility as far as rearrangement of its components is concerned. In a glass, although there is a random arrangement of the silica tetrahedra and of the alumina groups, there is nevertheless a certain rigidity toward free movement of the molecules not experienced in gels. Likewise, the diffusion of the reacting ions through the gel mass is much greater than can be obtained through the solid glass; this should make considerable difference in the time of reaction and also perhaps in the end product. Furthermore, these reactions go beyond those of Noll's by introducing into the investigation the effect of time, degree of comminution and type of glass.

Results

Montmorillonite was found to be formed in acid, neutral and basic solutions. The following table shows the conditions under which the clay was formed. As stated above, all reactions were carried out at 300°C.

Table 1

Reagent	Ratio $R_2O:Al_2O_3$	Time (Hours)	Product
H_2O		100	Montmorillonite et al.
H_2SO_4	0.2:1	50	Montmorillonite
H_2SO_4	2:1	143	Montmorillonite
$Ca(OH)_2$	0.2:1	40	Montmorillonite
$Ca(OH)_2$	2:1	144	?
NaOH	0.2:1	151	Montmorillonite and kaolin
NaOH	2:1	144	?
NaOH	4:1	148	Montmorillonite
KOH	0.2:1	149	Montmorillonite?
KOH	1:1	148	Montmorillonite et al.
$Mg(OH)_2$	0.2:1	64	Montmorillonite
$Mg(OH)_2$	0.2.1	80	Montmorillonite
$Mg(OH)_2$	0.2:1	149	Montmorillonite
$Mg(OH)_2$	2:1	45	Montmorillonite

* For further details see H. H. Reynolds, Sc.D. *Thesis*, M.I.T. 1939.

Effect of Reagents

In all cases the products were of the non-swelling type of montmorillonite and ranged in color from white to grey and brown. Montmorillonite was formed in all of the alkali solutions, with the greatest latitude of concentration to be found with $Mg(OH)_2$, which is in accord with the findings of Noll. Sulfuric acid showed a greater latitude than the alkalis except for $Mg(OH)_2$; in particular, the products from the acid runs showed a high dye-adsorptive capacity for methylene blue. When obsidian was used, it was found that the natural glass was very stable and in alkali solutions yielded a product similar to orthoclase on heating for 150 hours, but no montmorillonite at these lengths of time. However, when the glass was reacted for 350 hours with sulfuric acid in an acid: alumina ratio of 2:1, it was found that a montmorillonite whose particles were not colloidal was formed, which had a dye-adsorptive capacity comparable to that of a Fuller's earth used in standard oil bleaching operations.

The formation of montmorillonite in both alkali and acid solutions corresponds to the metamorphosis of the glass in nature. In the case of the acid transformation, the reaction may have taken place in part in the air during periods of volcanic activity during which time the percentage of corrosive gases would have been quite high. The transformation could also take place in situ after the deposition of the glass or ash by the leaching action of slightly acid or alkaline solutions. However, the concentrations employed in these experiments do not necessarily give a comparison with the concentrations of the reagents in nature, since the higher concentrations used in this work may act simply to increase the rate of reaction. In nature, a lower concentration of the acidic or basic solution may accomplish the same effect in a longer time. Whether the swelling properties and especially the particle size of montmorillonite and particularly Fuller's earths are due to the formation of the clay under different conditions cannot be stated at present. It may be that the reaction takes a different course under different conditions; the non-colloidal montmorillonite may be merely a stage in the formation of a colloidal clay. If one reaction takes place through the intermediate formation of gels, whereas the other is a process of devitrification and hydration, different end products might be expected. In any event, the results of these experiments seem to indicate that Fuller's earths may be found in acidic leaching solutions.

Effect of the Type of Glass

The obsidian was found to be much more stable than the synthetic glass having a composition comparable to that of the Wyoming bentonite. This is quite in accord with the findings of Königsberger and Müller (9), who reported obsidian to be extremely stable under hydrothermal conditions. This stability is probably due to the high silica content, since it is necessary first to remove the excess of silica from the glass before the alteration can be complete. The $Al_2O_3:SiO_2$ ratio in the glass is 1:9, while in the montmorillonite this ratio is 1:4 or 1:5. The silica is presumably transported either as a solution or in the form of a silicic acid sol. The two types of glasses used in these experiments represent the extremes of alumino-silica glasses found in nature.

Effect of Time

To determine the effect of time on the reaction, runs were made under similar conditions of reagent, temperature and pressure, but for varying lengths of time. It was found in every case, that whereas montmorillonite could be formed in 150 hours, heating for 290 hours under the same conditions gave a product which could no longer be identified as montmorillonite. With $Mg(OH)_2$ solutions and synthetic glass, montmorillonite was formed at 64, 80, and 149 hours, but not with 289 hours heating. These results indicate that montmorillonite is not the end-product of the reaction, but is an intermediate step. That montmorillonite is comparatively stable, however, is shown by the fact that it can be produced over a considerable time range; if it were unstable, the period of time during which it could be detected would be more critical. These facts lead to the conclusions that either the bentonite beds are still in process of transformation, that the leaching action has ceased to be of any consequence, that the conditions necessary to effect the change are no longer present, or that the present rate of reaction is too slow to be detected at present. Inasmuch as these reactions took place at 300°C., it is probable that the reduction in temperature in bentonite beds has so reduced the reaction rate that the bentonite is stable at present earth temperatures, although the reactions may be still proceeding at extremely low rates. In fact, it is generally conceded by geologists that the alteration of the glass usually took place at temperatures not much higher than the boiling point of water.

Effect of Comminution

The effect of particle size of the glass was investigated by making several runs with glass of 200 mesh particle size and similar runs with

glass ground to about 10 microns. It was found that the size of the reacting glass has a considerable effect on the reaction. Whereas montmorillonite was formed with the 200 mesh glass on heating for 150 hours, the finely ground glass gave an x-ray pattern which could not be identified. All the runs with the finely ground glass, regardless of the reagent used, showed the same x-ray pattern.

These facts indicate that the alteration, at these temperatures at least, is a surface phenomenon, and not merely an inter-molecular rearrangement. Presumably the surface of the glass is attacked, causing the alteration of the glass at that point. The reaction then proceeds through a hydration and adsorption of ions onto the crystal lattice of the clay particle, the rate of reaction of the interior of the glass particle depending to a certain extent upon the diffusion of the reagent through the reacted glass. It seems likely that the reaction is partially a devitrification phenomenon, in which the alumina and silica groups rearrange under the influence of temperature to give a crystalline structure. The alteration of glass to montmorillonite is probably a combination of the chemical reactions attendant with leaching, and a devitrification of the glass. The relative magnitude of these effects depends to a certain extent upon the stability of the glass.

General Discussion

The montmorillonite produced in acid solution was brown in color and had a high adsorptive capacity; this is no doubt a form of Fuller's earth, and indicates that this type of clay may have been formed from acid waters acting on volcanic glass. None of the montmorillonite produced showed swelling. There may be several reasons for this. In the alteration of the glass from the random network of silica tetrahedra which Warren and Biscoe (17) have shown to exist in a glass, to the three-layer lattice of montmorillonite, it is possible that these three-layer stacks were imperfectly formed and were still joined together by oxygen bonds. Relatively few such bonds would be required to prevent swelling, yet it is probable that such an alteration of the structure would not appear in an x-ray diffraction pattern. Furthermore, the high temperature and high pressure treatment might have so distorted the lattice that swelling was impossible. By employing different conditions in this type of reaction, it should be possible to obtain a swelling montmorillonite from glass.

These experiments have shown that montmorillonite can be produced from glass under the conditions shown above. It is recognized, however, that an analogy between conditions of these experiments and the conditions existent in geologic times must not be pushed too far. Mont-

morillonite or bentonite beds were formed at temperatures considerably below 300°C. While the effect of an elevated temperature is an enhanced rate of reaction, it may also result in a different type of reaction. So that the fact that montmorillonite may be produced at 300°C. does not necessarily mean that the same conditions would yield montmorillonite at 150°C. However, these reactions may have a value much beyond their significance to geologists.

Inasmuch as clays differ widely, even in the same locality, it would be of considerable importance to be able to produce a clay of known characteristics. Particularly in countries where such clays are scarce or nonexistent and must be imported at some cost, as in Rumania for example, it is conceivable that a method of manufacturing clay from its constituents, silica and alumina, may be of considerable value. Since silica and alumina are common and cheap materials, considerable expense could be incurred in such a process and still show a profit over transportation charges. In addition, it should be possible to produce clays of any desired properties by using appropriate means, so that one could tell, a priori, what type of clay he would obtain from certain materials under certain conditions. Where a small amount of clay of given characteristics was desired in a certain industry, this method should prove quite feasible.

It is proposed to continue this work under different conditions. Since we can simulate nature in all her effects except time, it is desired to compensate for this by the accelerating effect of other factors. As pointed out above, elevated temperatures offer certain definite objections. It is now proposed to carry out this alteration of glass at lower temperatures, but to circulate the leaching solutions at rates enormously faster than occur in nature. In this manner, the glass will be continually attacked by new solution, which corresponds to natural conditions, and laboratory results should be entirely comparable to those obtained in nature.

Summary

It has been shown that montmorillonite can be produced by high temperature and high pressure alteration of both a synthetic glass having a composition of that of montmorillonite, and a natural obsidian under acid, neutral and basic conditions. The conditions have much more latitude in the case of $Mg(OH)_2$ than with the other alkalis. The obsidian was found to be very stable and yielded a Fuller's earth only after fifteen days' heating. The montmorillonite produced was nonswelling, and that produced in acid medium showed the characteristics

of Fuller's earth. Though admittedly, conditions did not simulate geologic conditions, nevertheless, the conditions of formation at these temperatures have been indicated. Attention has been called to the possibility of using this method to produce clays of given characteristics synthetically in countries where natural clays are scarce, or in those industries where a highly specialized clay is desired in relatively small quantities.

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SANTA LUZIA DE GOYAZ METEORITE

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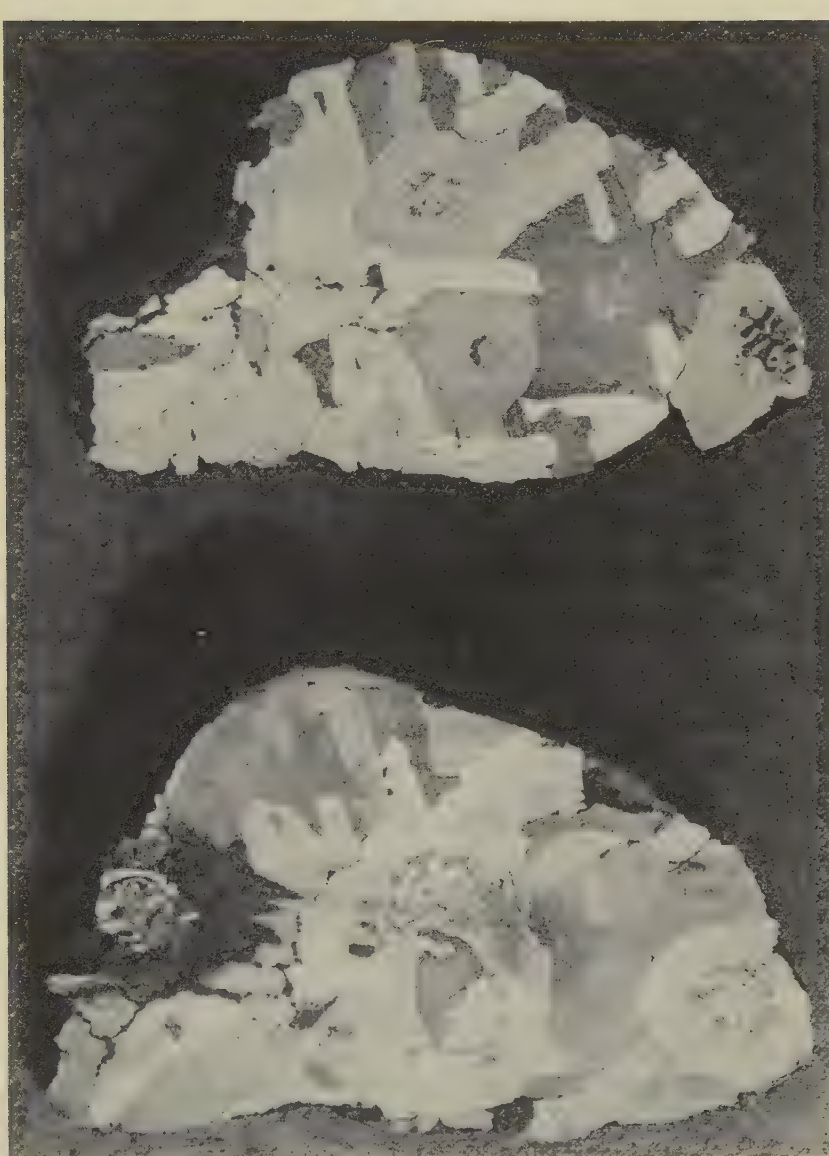
The Santa Luzia de Goyaz meteorite has been partially described by Oliveira (1931) who gives a chemical analysis and history for the iron and says that the structure is octahedral. Mention of it has also been made by Araujo (1931), Vidal (1931), and Betim (1935). The writer has made a study of this meteorite and finds a much different chemical composition from that published by Oliveira and in addition believes that the structure is of considerable interest.

History

From the files of the United States National Museum and from abstracts (*Mineralogical Abstracts*, vols. 5 and 6) of the above papers, the writer has compiled the following brief outline of the history of the Santa Luzia de Goyaz meteorite. It was found in 1927 (Oliveira gives the date as 1921) in the Negro Monte ravine, on the Paiva estate about 20 kilometres from the city of Santa Luzia, Goyaz, Brazil. Six fragments, whose total mass is 1923 kilograms, are known. The largest of these (1890 kilograms) is in the National Museum in Rio de Janeiro, Brazil. The United States National Museum has 22 kilograms in its collection.

Description

An etched surface of a slice from one of the individuals shows a remarkable structure (Figs. 1 and 2). There are roughly rectangular areas of kamacite up to 3.5 cm. by 1 cm. which are free of visible impurities. Intermingled with these rectangles are more or less circular areas of kamacite which are up to 5 cm. in diameter with irregular cores of schreibersite and troilite. The kamacite, surrounding these cores is, in general, almost 1 cm. thick. The specimens shown in Figs. 1 and 2 are cut from the same individual and are approximately 1.5 cm. apart. Some idea of the coarseness of the structure is obtained by observing the development of the small area of troilite in the lower centre of Fig. 1 into the large area of troilite in the lower centre of Fig. 2. There is a little taenite between grains of kamacite and there are traces of plessite at some of the boundaries of schreibersite.



FIGS. 1 and 2. Santa Luzia de Goyaz, coarsest octahedrite: Etched slices showing the large development of kamacite crystals, troilite and schreibersite. Mag. 4/5.

Chemical Analysis

Two small pieces were cut from the mass and the surfaces ground away to provide only fresh material for chemical analysis. One piece (about 18 grams) was from the average kamacite and taenite mass and contained a little plessite. The analysis of this specimen is given in Table 1, column 1. The other specimen (about 16 grams) was cut from a kamacite

Table 1. The Santa Luzia de Goyaz Meteorite: Chemical Analyses

	1[1]	2[1]		3[2]
		a	*b*	
Fe	92.81%	93.88%	93.45%	95.33%
Ni	6.48	6.14	6.11	2.64
Co	0.348	0.096	0.096	0.42
Cu	0.043	0.079	0.079	0.22
P	0.325	0.240	0.239	0.39
S	0.004	trace	trace	—
C	0.014	0.006	0.006	0.96 (C + Pt, etc.)
Pt, etc.	0.023	0.023	0.023	
Cr	nil	nil	nil	—
Total	100.05%	100.46%	100.00%	99.96%

[1] Analyst: V. B. Meen.
[2] Analyst: A. Girotto.

1. Average sample of kamacite and taenite mass.
2. *a*. Kamacite area free of taenite but contaminated by a little schreibersite.
   b. Analysis 2*a* reduced to the sum of 100
3. Analysis of average mass given by Oliveira (1931).

area and, so far as could be ascertained, did not contain any taenite or plessite. It did, however, contain a little schreibersite. The chemical analysis of this kamacite is given in column 2*a* and reduced to the sum of 100 in column 2*b*. In both analyses, iron was determined volumetrically and nickel was determined on separate portions in which the iron was held in solution by tartaric acid. The analysis by A. Girotto, published by Oliveira (1931), is given in column 3 and is assumed to be of the average kamacite-taenite mass. The chief differences between the present analyses and the older one are the values for iron and nickel. Oliveira's value for nickel is extremely low.

The chemical composition of kamacite has long been a matter of interest. It is only when coarsely crystallized irons are discovered, in which the kamacite can be separated from the other constituents, that more information on this score may be obtained. The extremely coarse structure of the Santa Luzia de Goyaz meteorite afforded another such opportunity.

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NOTES AND NEWS

MOUNTING AND REMOUNTING DETRITAL MINERAL GRAINS ON SLIDES

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Repairing Old Slides

At the petrographic laboratory of the University of Kansas, it was found that a reference set of detrital mineral slides, purchased from a British firm some twelve years ago, had become unusable through deterioration. In many the balsam was badly cracked and discolored, and in some the cover glasses had separated and fallen off. A search of the standard manuals found instructions only for repairing thin sections,[1] hence it was found necessary to work out some modified procedure applicable to detrital mineral mounts. Three possible methods suggested themselves: (1) Mechanical separation and remounting of the grains. This would entail separation and disintegration of the original mount, removal of mineral grains by hand picking, and transfer to a new slide. (2) Chemical separation of grains and remounting. This would involve removal of old balsam by solution in xylol, or by treatment with some chemical, such as acetone, leaving the residual mineral grains available for remounting. (3) Treatment of the original mounting material to restore its serviceability. The latter method was selected as most promising of convenience and rapidity, and the following procedure was evolved through trial and error. It is passed on with the hope that other geologists may be spared the loss of time and materials entailed by experimentation.

1. First, the cover glass, if not already separated, is wedged off with a needle point or knife edge. The entire mount may adhere either to the object glass or to the cover glass, and in either case the subsequent treatment is the same. If part adheres to one and part to the other, that part on the cover glass is scraped off onto the object glass. In any event, however, as much as possible of the deteriorated balsam around the edges of the mount is chipped off with a needle point and discarded.

2. The object glass or cover glass carrying the mineral grains and adhering balsam is next placed on a hot plate at a temperature just sufficient to slowly melt the balsam. Either an adjustable electric hot plate, or a metal plate heated by a microbunsen burner, may be used. Temperature may be tested with a small lump of solid balsam on a separate object glass.

3. If bubbles form as the balsam is heated, they are dispelled by

[1] Holmes, Arthur, *Petrographic Methods and Calculations*, Murby, London, 1930, 243.

cautiously applying droplets of xylol from a wire, glass rod, or capillary tube. In general, the xylol should be added at the *edge* of the balsam, as otherwise the latter tends to be spread out over the slide too far. Following clarification, more balsam, in xylol solution, is added if necessary, and the bubbles dispelled as before if a new crop appears.

4. The procedure is completed by carefully lowering a clean cover glass, previously heated, over the mount, instantly withdrawing the slide, and allowing it to cool. When cool, the balsam should still be soft enough to receive a dent from a needle point without cracking. With practice, the entire operation outlined above, may be completed in 5 to 10 minutes per slide.

Making Multiple Mounts

The recent introduction of various synthetic resins having refractive indices up to 1.71[2] makes possible the permanent mounting of minerals in different media on the same slide. This is convenient for several purposes:

1. Mounting light and heavy mineral crops from the same sample on the same slide, each in a medium of index most suitable for study of its particular composition. In this way the minerals from each sample are kept together, and duplicate indexing and filing are avoided.

2. Mounting portions of the same heavy mineral crop in different media on the same slide. Identification of the minerals in multifarious assemblages may be facilitated by study of relative relief and indices in mounting material representing well-spaced points on the refractive index scale. By having the different mounts on the same slide, comparison is made easier and more rapid. In preparing slides for this purpose, the heavy mineral concentrate is split into two or three equal and representative fractions, and these are mounted side by side. Mineral counts may be made in the usual way for each mount, and the results totalled.

3. Preparing reference or study sets for students. For this purpose, grains of the same mineral may be mounted on the same slide in two or three different media. Grains of assorted sizes and shapes are preferable, and for the more common detrital minerals these may be obtained by hand picking from screened sands under the binocular microscope. A selection is made from each fraction of each sample. An alternative method for minerals less readily available in this way is to crush and

[2] Cameron, E. N., Notes on the synthetic resin hyrax: *Am. Mineral.*, **19**, 375–383 (1934).
Keller, W. D., A mounting medium of 1.66 index of refraction: *Am. Mineral.*, **19**, 384 (1934).
Alexander, A. E., Recent developments in high index resins: *Am. Mineral.*, **19**, 385 (1934).

screen a larger specimen of the desired mineral. A few grains from each screen below 0.5 mm. are used for each mount. These of course will fail to show typical shapes and textures of water-worn grains, but will display characteristic optical properties.

For making multiple mounts, slides 1 in. by 3 in. are preferable. The object glass is placed on a hot plate, the mineral grains are added at two or three separate spots, and the mounting material applied. If necessary, an even distribution of the grains may be effected by applying one or more drops of xylol. With two mounts on a slide, the procedure is simple, but with three more care is required. As an example, it may be assumed that a given mineral or mineral assemblage is to be mounted in: (a) balsam or kollolith[3] (index c. 1.54), (b) aroclor 4465[4] (index c. 1.66), and (c) hyrax[5] (index c. 1.71). These media are applied in the order of

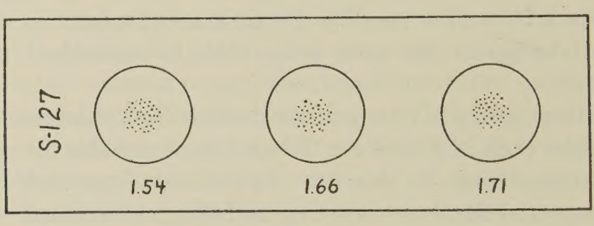

FIG. 1. Diagram of slide with three mounts, natural size.

decreasing melting points, hyrax first and aroclor last. Hyrax may be used satisfactorily in the solid form; for supplementary discussion, however, the reader is referred to the paper by Cameron. If bubbles develop, they may be removed by treatment with drops of xylol. In this event, however, care must be taken to avoid excessive spreading of the material, and to make certain that the xylol is all driven off, if the maximum refractive index is desired. Balsam may be used in either solid or dissolved form, the latter being less likely to develop bubbles. Aroclor 4465 is used in solid form, and causes no difficulty with bubbles. When all three mounts are ready, cover glasses are placed on them and the slide is withdrawn and allowed to cool. The final step consists in labelling the slide, and this is best done with a diamond marking pencil. In addition to the regular serial number or name, it is desirable also to place under each mount its refractive index. The completed slide will have the appearance shown in Fig. 1.

[3] Obtainable from Voigt & Hochgesang, Göttingen, Germany.

[4] Described by Keller, *op. cit.* Obtainable from Monsanto Chemical Company, St. Louis, Mo.

[5] Described by Cameron, *op. cit.* Obtainable from Eimer and Amend, New York City.

NOMINATIONS FOR OFFICERS OF THE MINERALOGICAL SOCIETY OF AMERICA FOR 1940

The Council has nominated the following as officers of *The Mineralogical Society of America* for the year 1940:

PRESIDENT: William F. Foshag, U. S. National Museum, Washington, D. C.

VICE-PRESIDENT: Ian Campbell, California Institute of Technology, Pasadena, California.

SECRETARY: Paul F. Kerr, Columbia University, New York, N. Y.

TREASURER: Waldemar T. Schaller, U. S. Geological Survey, Washington, D. C.

EDITOR: Walter F. Hunt, University of Michigan, Ann Arbor, Michigan.

COUNCILLOR (1940–43): D. Jerome Fisher, University of Chicago, Chicago, Illinois.

The twentieth annual meeting of the Society will be held in Minneapolis, Minnesota, December 28–30, 1939, in connection with the 51st meeting of the Geological Society of America. The headquarters of the Geological Society will be at the Hotel Nicollet, and the scientific sessions and social functions will be held there.

Members of the Society who are planning to present papers at the scientific sessions of the annual meeting should notify the Secretary as soon as possible in order to receive the proper blanks for their abstracts. All abstracts should be in the Secretary's office by *November 1st.*

Advance announcement of the annual meeting will be distributed to members of the Society, along with the ballot for officers, in the middle of October. The final program of the meeting, including the schedule of papers, abstracts, and other information will be sent to each member with the December issue of *The American Mineralogist.* Further specific information regarding the annual meeting may be obtained from the Secretary's office.

PAUL F. KERR, *Secretary*

PROCEEDINGS OF SOCIETIES

Academy of Natural Sciences, Philadelphia, Pa.

A meeting of the Philadelphia Mineralogical Society was held on April 6, 1939, with president Harry W. Trudrell in the chair. Thirty-five members and twenty-five visitors were in attendance.

Mr. Martin Ehrmann addressed the society on *jade.* He described the various minerals which are accepted as true jades and those which are substituted for the more costly varieties. The present supply of jade comes mostly from Burmese river gravels, and is marketed at Canton, China, at an annual auction. The speaker showed a series of pictures of beautifully carved jades and discussed the Chinese mythology and symbolism involved in their carving and interpretation.

A meeting of the Philadelphia Mineralogical Society was held on May 4, 1939, with fifty members and thirty-five visitors in attendance.

Mr. John C. Boyle spoke to the society of his recent trip through Europe, where he visited numerous mineral localities and museums. He described and illustrated with beautiful pictures various interesting features, and showed specimens of some of the unusual dike rocks of the Oslo region, also various other mineral specimens.

Mr. Ralph Hoffa showed a ball of smoky quartz which he himself had cut and polished. Mr. Arthur Guest, Jr., exhibited torbernite, uranophane and hyalite from the Mermaid Lane quarry in Philadelphia.

The June meeting was held on June 1, 1939, with forty-two members and eighteen visitors in attendance.

The deaths of Mr. Bentley Morrison and Dr. Witmer Stone were announced and resolutions of sympathy were sent to Mr. Morrison's mother, and to the wife of Dr. Stone.

Mr. Louis Moyd spoke on the mineralogy of metamorphosed limestones, discussing the phenomena associated with the alteration of limestones by granitic magmas and emanations. Among the matters discussed were the origin of the materials forming the contact minerals found in marbles, their paragenesis, and a summary of the metamorphic facies theory. Also discussed was the effect of limestone on small, cross-cutting igneous bodies. Here were described the development of sphene, diopside, scapolite and other minerals in otherwise normal granitic pegmatites. The speaker spoke of the assimilation of limestone by larger igneous masses, discussing the development of the carbonate-containing silicates, and the evidence for and against primary calcite. He developed the theories which advocate the origin of alkaline rocks through desilication of granitic magma, showing how basic silicates might form, thus enriching the still fluid magma in alkalies and alumina, and how these materials could be concentrated by upward streaming of volatiles. The speaker favored the origin of some corundum and spinels by desilication and concentration of alumina. To point out the application of many of the theories discussed, the speaker described the apatite-pyroxenite locality of Palabora, in the Transvaal, and the well-known Haliburton-Bancroft area in Ontario. The discussion was illustrated by many specimens, lantern slides and maps.

LOUIS MOYD, *Secretary*

Dr. William Arthur Tarr, professor of mineralogy and economic geology at the University of Missouri, died on July 28. He was fifty-eight years old.

Dr. Alfred Harker, emeritus reader in petrology at the University of Cambridge, a fellow of St. John's College, died on July 29 at the age of eighty years.